Effects of Strain Rate and Temperature on Fracture Toughness and Tensile Properties of CPVC

BY

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عمادة الدراسات العليا
DEANSHIP OF GRADUATE STUDIES

To my mother

To my wife

To my son and family members

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The author would like to acknowledge the advice, support and encouragement which was bestowed upon him during the course of this research.

First and foremost, all sincere praises and thanks are due to our God, Allah (SWT), who created us and guided us to this great religion, i.e. Islam and who alone made this accomplishment possible. I seek His mercy, favor and forgiveness. No one can thank God as he deserves because of his limitless blessings on us.

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NOMENCLATURE

a	Crack length (m)
B	Specimen thickness (m)
ε	Engineering strain
E	Elastic modulus (MPa)
K	Strength coefficient (MPa)
K_c	Fracture toughness (MPa.m$^{1/2}$)
K_{IC}	Plane strain fracture toughness (MPa.m$^{1/2}$)
K_Q	Provisional fracture toughness (MPa.m$^{1/2}$)
L_f	Length of fracture (m)
L_i	Original length (m)
n	Strain-hardening exponent
P	Load (N)
P_Q	Critical load (N)
R	Plastic deformation zone length (m)
S	Loading span (m)
T	Temperature (^{o}C or K)
W	Specimen width (m)
v	Crosshead speed (mm/min)
σ_{ys}	Yield stress (MPa)
$\dot{\varepsilon}$	Strain rate (s^{-1})
ε_y	Yield strain
ε_f	Fracture strain

LIST OF ABBREVATIONS

COD	Crack opening displacement
CPVC	Chlorinated poly vinylchloreide
CTOD	Crack tip opening displacement
DSC	Differential scanning calorimetry
HDPE	High density polyethylene
LEFM	Linear Elastic Fracture Mechanics
PA	Poly acetal
PC	Poly carbonate
PE	Poly ethylene
PEEK	Poly etherether ketone
PEI	Poly etherimide
PEK-C	Phenol phthalein polyether ketone
PET	Poly ethylene terethphthalate
PMMA	Poly methylmethacrylate
PP	Poly propylene
PS	Poly styrene
PVC	Poly vinylchloride
SEN	Single edge notch
SENB	Single edge notch bending
T_g	Glass transition temperature
TPX	Poly 4-methyl-1-pentene
uPVC	unPlasticized PVC

THESIS ABSTRACT

NAME: **TURKI ABDUL-AZIZ AL-QAHTANI**

TITLE: **EFFECTS OF STRAIN RATE AND TEMPERATURE ON FRACTURE TOUGHNESS AND TENSILE PROPERTIES OF CPVC**

DEPARTMENT: **MECHANICAL ENGINEERING**

DATE: **22 OCTOBER, 2005**

In recent years, there has been a large increase in the use of polymers in engineering applications. Along with this increase use will almost assuredly come failures. Chlorinated-PVC (CPVC) is a material designed to withstand higher temperature than PVC. For this reason, understanding the mechanical properties under different loading rates and temperatures has become increasingly important.

In this thesis work, strain rate (crosshead speed) and temperature dependence of fracture toughness and tensile properties of CPVC pipe material is investigated. Single-Edge-Notch Bending (SENB) and tensile specimens are used for fracture toughness and tensile tests, respectively. Five temperatures (-10, 0, 23, 50 and 70^{o}C) and three crosshead speeds (5, 50 and 500 mm/min) are considered in this study.

The results showed that both ductile and brittle fracture modes are possible. At 50^{o}C, brittle to ductile transition behavior is observed. A reduction in temperature and an increase in crosshead speed have favored brittle fracture. The yield stress and elastic modulus decreased with decreasing crosshead speed and increasing temperature. However, the value of fracture strain remained almost constant with crosshead speed at room temperature and below. At 50 and 70^{o}C, an enhancement of fracture strain occurred with increasing crosshead speed between 5 and 50 mm/min then stabilized with further increasing crosshead speed.

The value of fracture toughness increased with crosshead speed between 5 to 50 mm/min for all temperatures. However for the range of 50 to 500 mm/min, it reduced slightly at low temperatures (-10, 0 and 23^{o}C) and enhanced at high temperatures (50 and 70^{o}C) with increasing crosshead speed. It increased with temperature then it dropped at 70^{o}C for all crosshead speeds. The crosshead speed and temperature effect is explained in terms of the crack tip blunting, crack tip plastic zone and fracture surface condition. Moreover, the relationships between fracture toughness and toughness modulus were explained.

MASTER OF SCIENCE DEGREE

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خلاصة الرسالة

الاسم : **تركي عبدالعزيز القحطاني**

العنوان: **تأثير متغير الشد ودرجة الحرارة على متانة الكسر و خواص التوتر في (CPVC)**

القسم : **الهندسة الميكانيكية**

التاريخ: **22 أكتوبر 2005م**

في الأعوام الحديثة, هناك ازدياد واسع في استخدام البوليمرات في المجالات الهندسية. ومع تزايد هذه الاستخدامات سوف يحدث بالتأكيد الأجهادات. كلورايد البولي فينيل المكلور (CPVC) هي مادة صممت لتحمل درجة حرارة أعلى من كلورايد البولي فينيل (PVC). لهذا السبب, فان فهم الخواص الميكانيكية تحت متغيرات الحمولة و درجات حرارة مختلفة أصبح متزايد الأهمية.

في هذه الدراسة, تم بحث اعتماد متغير الشد (سرعة رأس الجهاز) ودرجة الحرارة على متانة الكسر و خواص التوتر في مادة انبوب كلورايد البولي فينيل المكلور (CPVC). تم استخدام عينات حافة السن المثني المفرد (SENB) و التوتر في اختبارات متانة الكسر و التوتر على التوالي. خمس درجات حرارة (-10, 0, 23, 50 و 70 درجة حرارة مئوية) و ثلاث سرعات لرأس الجهاز (5, 50 و 500 مليمتر/ دقيقة) اعتبرت في هذه الدراسة.

أظهرت النتائج بإمكانية كل من أساليب الانكسار اللينة والهشة. عند 50 درجة حرارة مئوية, لقد شوهد سلوك التحول من الليونة إلى الهشاشة. إن نقصان درجة الحرارة و زيادة سرعة رأس الجهاز تستحسن الانكسار الهش. ينقص إجهاد الخضوع و عامل المرونة بنقصان سرعة رأس الجهاز و ازدياد درجة الحرارة. و لكن إن قيمة انكسار الشد بقي شبه ثابت مع سرعة رأس الجهاز عند درجة حرارة الغرفة وأقل منها. عند 50 و 70 درجة حرارة مئوية, حصلت زيادة في انكسار الشد مع ازدياد سرعة رأس الجهاز ما بين 5 و 50 مليمتر/ دقيقة ثم ثبتت مع زيادة أكثر لسرعة رأس الجهاز.

إن قيمة متانة الكسر زادت مع سرعة رأس الجهاز ما بين 5 و 50 مليمتر/ دقيقة لكل درجات الحرارة. ولكن في المجال ما بين 50 و 500 مليمتر/ دقيقة, لقد نقصت قليلاً عند درجات الحرارة المنخفضة (-10, 0 و 23 درجة حرارة مئوية) وزادت عند درجات الحرارة المرتفعة (50 و 70 درجة حرارة مئوية) مع ازدياد سرعة رأس الجهاز. لقد زادت متانة الكسر مع درجة الحرارة ثم هبطت عند 70 درجة حرارة مئوية لكل سرعات رأس الجهاز. إن تأثير رأس الجهاز ودرجة الحرارة قد شرحت بالمصطلحات التالية: رأس الشق غير الحاد, منطقة رأس الشق اللزجة و حالة سطح الكسر. علاوة على ذلك, لقد شرح العلاقات بين متانة الانكسار و عامل المتانة.

درجة الماجستير في العلوم
جامعة الملك فهد للبترول والمعادن
الظهران - المملكة العربية السعودية

CHAPTER 1

INTRODUCTION

1.1 Background

Polymers are materials composed of long chain-like molecules in which the atoms are bound together by strong covalent bonds. A polymer structure contains many (poly-) repeats of some simpler chemical units called *mer* or *monomer*. A monomer is also used to indicate the basic chemical compound with which the polymer is polymerized. Usually the biggest difference in polymer properties results from how the atoms and chains are linked together in space [1].

Polymers exist in nature in such forms as wood, cotton, silk, hair and flesh. In addition, there are countless man-made or synthetic polymers being used in more traditional applications such as clothing, packing and various household items. However, in recent years these synthetic polymers are being increasingly used in engineering applications related to the automotive and aerospace industries, civil engineering applications, gas and liquid transport systems, paints and coatings, and electronics industries. The interest in engineering polymers is driven by their manufacturability, recyclability, mechanical properties, and lower cost as compared to many alloys and ceramics [2].

Polymer can be divided into two types: thermosets and thermoplastics. A thermoplastic is a polymer that softens when heated (and eventually liquefies) and hardens again when cooled. Thermoplastics are commonly used in piping, valves, hoods, and storage tank linings. Typical thermoplastics used in pipes are polyvinyl chloride (PVC), chlorinated polyvinyl chloride (CPVC), polypropylene (PP), poly methylmethaacrylate (PMMA), poly etherether ketone (PEEK) and poly carbonate (PC), etc. Thermoplastics have a number of advantages including low cost, ease of fabrication, good chemical resistance, good strength to weight ratio and high durability [3].

1.2 Chlorinated Polyvinyl Chloride (CPVC)

Chlorinated polyvinyl chloride (CPVC) is one of the important polymers used nowadays in pipes and fittings. CPVC is produced by the post-chlorination of polyvinyl chloride (PVC). The effect of adding more chlorine to the PVC molecule is to raise the glass transition temperature (T_g) of the base resin to the 115-135^{o}C range. The extra chlorine imparts improved resistance to combustion and smoke generation, higher tensile strength and higher modulus [4].

PVC belongs to the family of vinyl polymers and is commercially produced mainly by addition polymerization process. After the completion of the process more chlorine is introduced into the bulk material. The chlorination of PVC is a free radical process, catalyzed by heat, ultraviolet radiation and radical initiators. Chlorine molecule Cl_2 splits into two free radicals {$Cl^{\cdot}$} under the application of heat. One free radical joins the PVC monomer by replacing one H atom to form CPVC. The other free radical combines with H atom to form HCL. All the monomers in the PVC chain are not chlorinated; usually every fifth or sixth monomer is chlorinated. This is done by controlling the quantity of chlorine. This is also desirable because high chlorine content deteriorates the mechanical properties of the product by making it brittle. For optimum results, chlorine is kept up to 56.5 % by weight [5, 6]. The effect of chlorine addition on some physical and mechanical properties is shown in Table 1.1. The result and discussion of tensile properties of CPVC (supplier-A) will be given in chapter 4. Experimental results for CPVC (supplier-B) and data from supplier for CPVC and PVC and are provided for comparison.

CPVC offers a blend of chemical and weather resistance, mechanical strength and low installed cost for diverse applications including pulp and paper, metal treating, fertilizer production, ore benefaction, pollution control and wastewater treatment. In fact, CPVC has been used in a variety of processes and industries for over 30 years [7].

Because of its excellent chemical resistance at elevated temperatures, CPVC is ideally suited for self-supporting constructions where temperatures up to 93^{o}C are present. It has a high strength-to-weight ratio and provides good electrical and thermal insulations [3].

Traditional applications of CPVC compound are hot and cold water distribution piping and fittings, as well as industrial pipe fittings and valves that can handle liquid chemicals. CPVC pipes can withstand pressures up to 1.40 MPa as compared to 0.95 MPa for PVC at 50˚C [1, 4]. A recent development is specialized CPVC pipe and fitting compounds for use in fire-sprinkler systems. The material exhibits excellent fire resistance, chemical resistance and is readily available in sheets, rods and tubing. It may be cemented, welded, machined, bent and shaped readily [3, 4].

Table 1.1 Comparison of physical and mechanical properties of CPVC and PVC.

Property (@ 23 ^{o}C)	CPVC			PVC
	Present Study (Supplier-A)	Experimental result (Supplier -B)	Data from Supplier-A [8]	Data from Supplier-A [8]
Yield Stress (MPa)	47	55	55	48
Elastic Modulus (GPa)	2.89	3.00	2.90	2.75
Poisson's Ratio	-	-	0.27	0.38
Compressive Strength (MPa)	-	-	70	66
Flexural Strength (MPa)	-	-	104	88
Impact Strength (ft-lb$_f$/in)	-	-	1.50	0.65
Max. Operating Temp. (^{o}C)	-	-	93	60
Glass Transition Temp. (^{o}C)	120	-	115-135	90
Specific Gravity (g/cm^3)	-	-	1.55	1.42

1.3 Tensile Properties of Polymers

The tensile properties of polymers are specified with parameters of yield stress, elastic modulus and fracture strain. For many polymeric materials, the simple stress-strain test is employed for the characterization of these parameters. The tensile properties are highly sensitive to the strain rate, temperature and chemical nature of the environment. The data derived from tensile tests is essential for material selection during design of engineering components.

Three typically different types of stress-strain behavior are found for polymeric materials, as represented in figure 1.1. Curve *A* illustrates the stress-strain character for a brittle polymer, which fractures while deforming elastically. Another polymer behavior is represented by curve *B* where the material initially deforms elastically followed by yielding and a region of plastic deformation. Finally, the deformation displayed by curve *C* is totally elastic; this rubberlike elasticity (large recoverable strains produced at low stress levels) is displayed by a class of polymers termed the elastomers [9, 10].

1.4 Fracture Behavior of Polymers

The fracture behavior of polymeric materials has only recently become a major concern, as engineering plastics have begun to appear in critical structural applications. In most consumer products made from polymers (e.g. toys, garbage bags), fracture may be an annoyance, but it is not a significant safety issue [11].

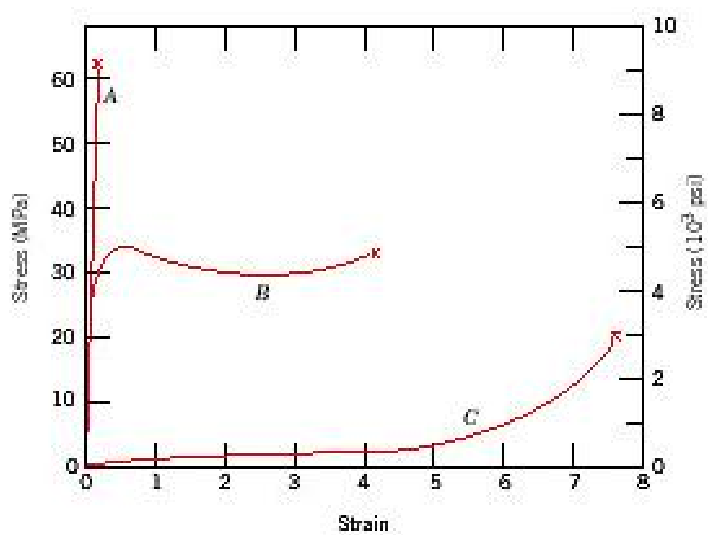


Figure 1.1 The stress-strain behavior for brittle (curve *A*), plastic (curve *B*) and elastomeric (curve *C*) polymers [9].

For thermoplastic polymers, both ductile and brittle fracture modes are possible, and many of these materials are capable of experiencing a brittle-to-ductile transition. Factors that favor brittle fracture are a reduction in temperature, an increase in strain rate, the presence of a sharp notch, increased specimen thickness and a modification of the polymer structure (chemical, molecular and/or microstructure). Glassy thermoplastics are brittle at relatively low temperature; as the temperature is raised, they become ductile in the vicinity of their glass transition temperatures and experience plastic yielding prior to fracture [9].

Deformation after the yield point may have various forms depending on the structure, mechanical and thermal history of the sample, such as shear yielding and cold drawing as depicted in figure 1.2. At an intermediate temperature between the brittle fracture and plastic deformation regimes many polymers undergo a non-catastrophic failure called crazing. Crazes form at highly stressed regions associated with scratches, flaws, dust particles and molecular inhomogeneities; they normally propagate perpendicular to the tensile stress axis. Associated with crazes are regions of very localized yielding, which lead to the formation of fibrils (regions wherein the molecular chains are oriented), and interspersed small voids that are interconnected [9, 12].

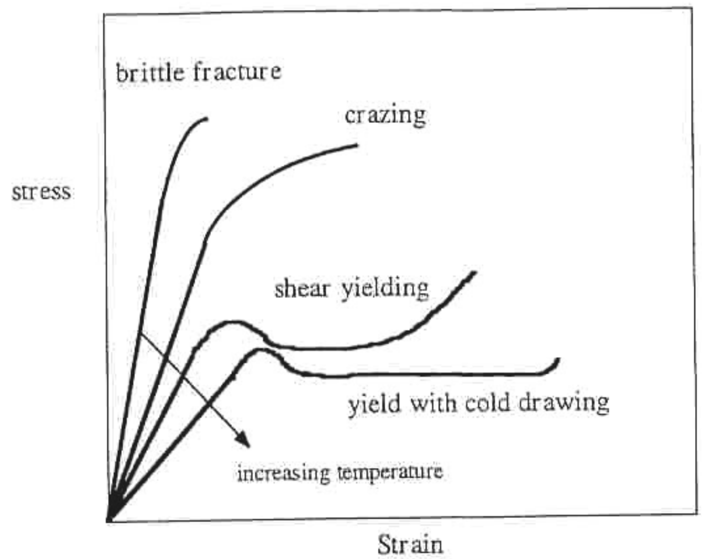


Figure 1.2   Schematic of some failure modes of polymers [12].

1.5 Fracture Toughness of Polymers

Fracture toughness is a measure of the ability of a material to resist the growth of a pre-existing crack or flaw. The design stress and crack size in combination with fracture toughness control the conditions for fracture. In order for the design engineer to make accurate predictions of the maximum allowable loads or crack sizes within the structure, it is essential that the predictions are based on a valid measure of fracture toughness. In other words, the accuracy of the predictions is highly dependent upon the validity of the fracture toughness value measured in the laboratory. In general, fracture toughness K_c may be expressed in the form:

$$K_c = Y\sigma\sqrt{\pi a} \tag{1.1}$$

where Y is a dimensionless parameter that depends on both the specimen and crack geometries.

Polymers have fracture toughness values in the range 1 to 5 MPa.m$^{1/2}$. Although these are low values, most polymers are used at low stresses due to their low ultimate strengths. Thus under typical usage, the likelihood of fracture is roughly similar to that of metals [13].

It is common experience that the resistance to cracking in many engineering materials depends on the rate of deformation, and the test temperature at which cracking takes place. Traditionally, the effects of rate and temperature have been interpreted in terms of simple stress-strain curves, tensile strain-to-fracture and stress/time-to-failure curves. Nowadays, however, such effects are given in terms of fracture toughness parameters and it can be studied either individually or in combination [14].

The factors that govern the fracture toughness of polymers include strain rate, temperature, tensile strength and molecular structure. At high rates or low temperatures polymers tend to be brittle, because there is insufficient time for materials to respond to stress with large-scale viscoelastic deformation or yielding [11].

Data on the fracture toughness versus temperature behavior of polymers are useful in selecting specific materials for service, as it is generally important to avoid high-stress use of materials at a temperature where its fracture toughness is low. Fracture toughness generally increases with temperature and usually decreases with higher rate of loading [13].

The fracture toughness can be measured in units of energy. The brittle material absorbs less energy, while a tough material would require a large expenditure of energy in the fracture process. For an unnotched tensile bar, the energy to break may be estimated from the area under the stress-strain curve. Maximum toughness is achieved with an optimum combination of strength and ductility [15].

1.6 Present Study

The fracture behavior of CPVC depends on the mechanical properties such as yield stress, elastic modulus and fracture strain. Investigation of the effects of strain rate (crosshead speed) and temperature on these tensile properties of CPVC pipe material represents the first objective of this study. The crosshead speeds and temperatures that will be considered are 5, 50 and 500 mm/min and -10, 0, 23, 50 and 70^{o} C, respectively. The second objective of this research is to investigate the effects of strain rate (crosshead speed) and temperature on fracture toughness of CPVC pipe material using single-edge-notch bending specimens. These effects will be explained by analyzing crack tip blunting, crack tip plastic zone and fracture surface. An attempt will be made to relate the fracture toughness value to the toughness modulus determined from simple tensile test.

The thesis is divided into six chapters. Chapter two discusses a review of the relevant literature describing the effects of strain rate and temperature on tensile properties and fracture toughness of many polymers. In chapter three, the experimental procedure including specimen preparation for tensile and fracture toughness tests, testing apparatus and programs will be presented. In chapters four and five, the effects of strain rate and temperature on tensile properties and fracture toughness will be discussed and analyzed, respectively. Finally, conclusions drawn from this study and recommendations for future research are presented in chapter six.

CHAPTER 2

LITERATURE REVIEW

This chapter covers the essentials of the relevant work done by different researchers on tensile properties and fracture toughness of polymers. Included in this discussion is the effect of strain rate and temperature on tensile properties. Next, the effect of strain rate and temperature on fracture toughness will be discussed. The summary of the literature review and status of the present problem are then presented.

2.1 Effect of Strain Rate and Temperature on Tensile Properties

The effect of strain rate and temperature on yield stress and elastic modulus in different polymers has been studied by Langford et al. [16], Mizutani [17] and Tordjeman et al. [18] for PMMA, Parvin and Williams [19] for polycarbonate (PC), Kitagawa [20] for Polyacetal (PA), PMMA, PVC and PC, Bronnikov et al. [21] for Polyethylene (PE), Polyethylene terethphthalate (PET), nylon 6 and nylon 610, Stokes et al. [22] for polycarbonate (PC), polyether-imide (PEI) and polybutylene terephthalate (PBT), Kim and Ye [23] for PEI, Fernando and Williams [24] for polypropylene (PP), Chan and Williams [25] for Polyethylene (PE) and Jia and Kagan [26] for dry-as-molded nylon 6. They found that the yield stress and elastic modulus decreased with increasing temperature and decreasing strain rate.

Povolo et al. [27] studied the stress relaxation of unplasticized PVC below yield point. The temperature dependence of elastic modulus was determined from measured stress-strain curves. It has been shown that the elastic modulus decreased with increasing temperature. The true stress-strain curves for uPVC, measured at different temperatures and at the strain rate of $6.6 \times 10^{-4}\ s^{-1}$ are shown in figure 2.1, while figure 2.2 shows the variation of elastic modulus with temperature.

Bauwens-Crowet et al. [28] measured the ratio of yield stress to temperature as a function of strain rate for PVC in the temperature range of -180 to 23^{o}C and strain rate of 10^{-5} to $10^{-1}\ s^{-1}$. They found that the yield stress increased with increasing strain rate and decreasing temperature.

Che et al. [29] used J-integral and crack opening displacement (*COD*) concepts to study the effects of specimen dimension, specimen configuration and test conditions (crosshead speed, temperature), over a range of crosshead speeds from 0.01 to 100 mm/min and temperatures from -40 to 60^{o}C, on the crack resistance behavior of PVC. They found that both yield strength and Young's modulus increased with increasing crosshead speed. Also, they have shown that the yield strength decreased linearly with increasing temperature, while the Young's modulus decreased first with increasing temperature, then afterwards stayed relatively constant between -20 to 40^{o}C, then decreased again. The Young's modulus and yield strength are shown in figure 2.3(a) as a function of crosshead speed at room temperature and in figure 2.3(b) as a function of test temperature at a crosshead speed of 1 mm/min.

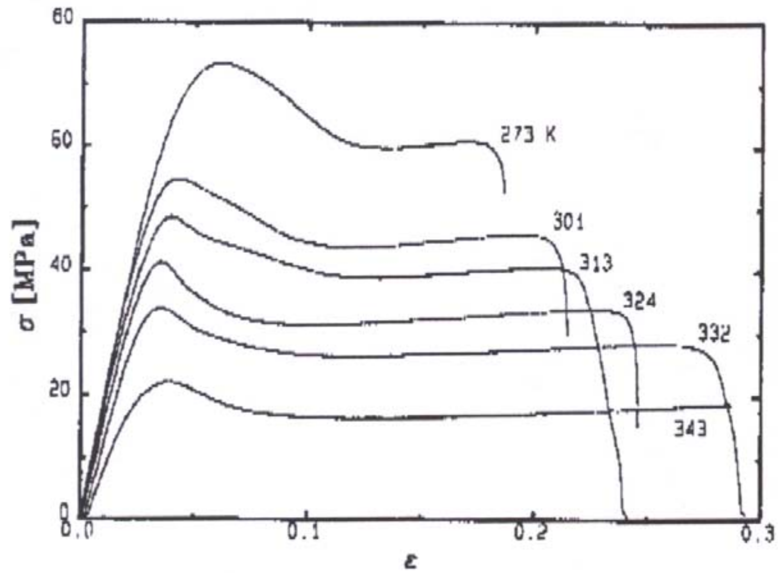


Figure 2.1 True stress-strain curves for unplasticized PVC at different temperatures and at the strain rate of 6.6 x 10^{-4} s^{-1} [27].

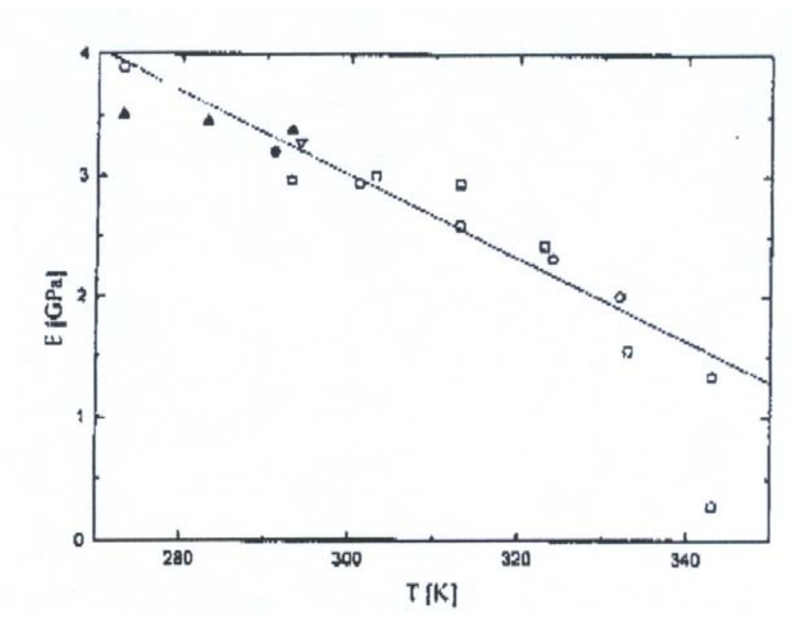


Figure 2.2 Variation of elastic modulus with temperature for PVC at $\dot{\varepsilon}$ =6.6 x 10^{-4} s^{-1}[27].

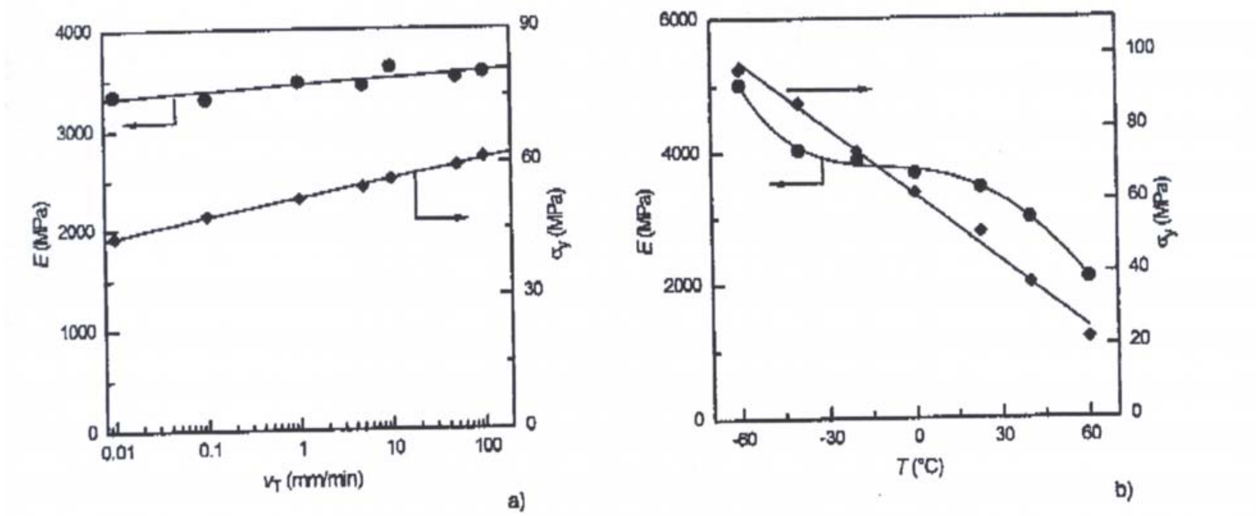


Figure 2.3 Tensile properties in dependence of (a) crosshead speed at room temperature and (b) test temperature at a crosshead speed of 1 mm/min for PVC [29].

Hitt and Gilbert [30] studied tensile properties of unplasticized PVC at elevated temperatures. The temperature range used is from 23 to 180^{o}C. It was found that the stress at break decreased steadily with increasing test temperature.

Green et al. [31] developed tensile and compressive failure diagrams for two thermoplastics mixtures, PVC and PET (poly-ethylene-terephthalate). They have shown that the tensile strength decreased with increasing temperature and decreasing strain rate for both PVC and PET. Their results are reported in figure 2.4, where tensile strength for PVC is plotted vs. temperature for three different strain rates. At each strain rate, there was a transition temperature below which failure is brittle and above which failure is ductile. Brittle-ductile transition temperatures estimated by tensile failure mechanism diagrams predicted a processing window of temperatures and strain rates in which PVC should be failing in the brittle mode and PET should be failing in the ductile mode.

Vincent [32] studied the effect of fracture strain for PMMA as a function of temperature. He found that the fracture strain remained constant in the temperature range of -180 to 23^{o}C then as the temperature raised the fracture strain increased rapidly. Also, he showed the dependence of brittle-ductile transition temperature for PMMA and PVC over the range of strain rates (10^{-4} to 10^{4} s^{-1}). He found that the brittle-ductile transition temperature increased with strain rates.

Ye et al. [33] carried out tensile tests of TPX (poly-4-methyl-1-pentene) polymer at ambient temperature at various crosshead rates (from 0.5 to 500 mm/min). They found that the tensile yield stress and Young's modulus at ambient temperature increased with increasing crosshead rates. However, no yield behavior observed at v = 500 mm/min.

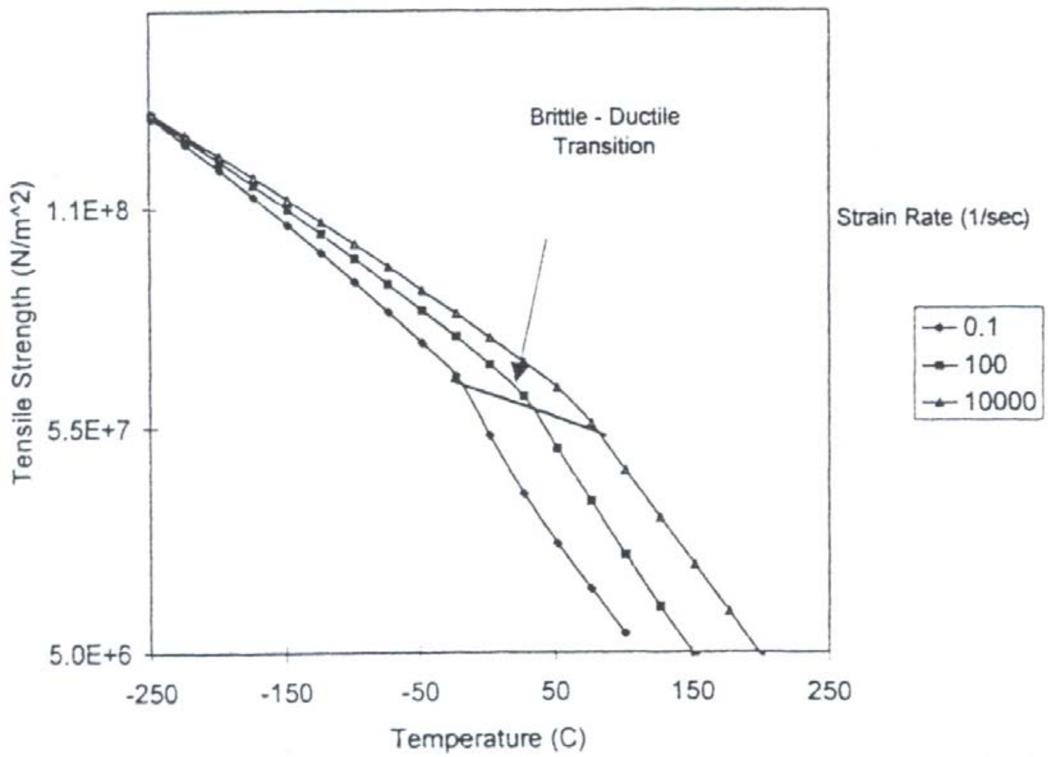


Figure 2.4 Tensile failure strength versus temperature for three different strain rates for PVC [31].

Saghir [34] studied the effect of temperature on monotonic properties of high density polyethylene (HDPE) in the temperature range -10 to 50^{o}C. He concluded that the yield stress and elastic modulus decreased with increasing temperature.

Merah et al. [35] investigated the effect of temperatures ranging from -10 to 70^{o}C on the mechanical properties of CPVC pipe fitting material. They found that the yield strength and elastic modulus decreased linearly with increase in temperature. However, the fracture strain increased with increasing temperature. Brittle fracture occurred at temperatures below room temperature while ductile fracture occurred at and above room temperature.

2.2 Effect of Strain Rate and Temperature on Fracture Toughness

Williams [36] investigated the effect of temperature on fracture toughness for PVC using single-edge-notch (SEN) tension on four different sheet thicknesses. He concluded that the fracture toughness decreased with increasing temperature in the temperature range of -200 to -120^{o}C. Thereafter, it slightly increased in the temperature range of -120 to 20^{o}C.

Mandell et al. [37] investigated the fracture toughness of unplasticized poly (vinylchloride) pipe materials in the temperature range of -120 to 80^{o}C and time to failure of 10^{-3} to 120 s. The specimens used were double-edge-notched (DEN) tension. The data showed that PVC shifted from ductile, notch-intensive behavior to brittle, linear elastic fracture mechanics (LEFM)-controlled fracture as the temperature or the time to failure decreased. Under brittle conditions, the fracture toughness was insensitive both to failure time and to temperature.

Parvin and Williams [19] showed the variation of fracture toughness as functions of temperature for polycarbonate (PC) using SEN specimens. They found that the fracture toughness with the thickness of 5 mm decreased in the temperature range of -140 to -70^{o}C. Then, it sharply increased in the temperature range of -70 to 50^{o}C.

The fracture toughness of poly (methyl methacrylate) (PMMA) has been measured over a wide range of temperatures by Mizutani [17]. The measurements of the fracture toughness were carried out on a tapered double cantilever beam (T-DCB) specimen at a crosshead speed of 0.5 mm/min. It was reported that the fracture toughness decreased with increasing temperature over the temperature range of -30 to 60^{o}C. In the range of 70 to 90^{o}C, the values of fracture toughness were slightly higher than that at 60^{o}C. It increased above 90^{o}C, reaching a maximum at T_g (107^{o}C) and then dropped off with increasing temperature.

Marshal et al. [38] investigated the effect of strain rate and temperature on fracture toughness of PMMA. They have showed that fracture toughness increased with crosshead rate in the crosshead range of 10^{-3} to 10^{2} cm/min. The fracture toughness at crosshead rate of 0.5 cm/min in the temperature range of -180 to 100^{o}C increased up to the temperature of -100^{o}C then it decreased. However, the fracture toughness at crosshead rate of 50 cm/min increased up to the temperature of -50^{o}C then it decreased.

Ye et al. [33] studied the fracture behavior of TPX (poly-4-methyl-1-pentene) polymer using compact tension (CT) and single-edge-notch bending specimens. They found that when the crosshead rates were increased from low (0.5 mm/min) to high (500 mm/min), the fracture toughness of TPX polymer first increased and then dropped sharply at around 500 mm/min, as shown in figure 2.5. The reduction of fracture toughness was explained by a reduced molecular mobility of the polymer around the crack tip or a reduced plastic flow zone at high crosshead rates and a low ductility of the polymer was expected. They found that the fracture toughness for SENB specimen at different crosshead rates (v = 0.5, 100 and 500 mm/min) was rather high at temperatures less than 40^{o}C for the low or moderate crosshead rates. However, the value of fracture toughness at high crosshead rates was found fairly low and almost constant below the temperature of 40^{o}C then increased and reached a maximum value at 60^{o}C and then decreased. The glass transition temperature was 81^{o}C. Figure 2.6 shows the influence of temperature on the fracture toughness at three different crosshead rates.

Karger-Kocsis and Friedrick [39] reported the temperature and strain rate effects on the fracture toughness of poly (ether ether ketone) (PEEK) and its short glass-fiber reinforced composites. They used compact tension (CT) specimens. Testing was performed as a function of temperature, strain rate and heat treatment. The results showed that the fracture toughness of both materials was highly dependent on these factors causing either brittle or ductile damage. The fracture toughness of PEEK at low crosshead speed (v = 1 mm/min) at room temperature was around 7 MPa.m$^{1/2}$ and it did not change very much when reducing the temperature down to -60^{o}C or increasing it up to 60^{o}C. A further increase in temperature finally reduced the fracture toughness to about 2 MPa.m$^{1/2}$ at 180^{o}C. At high crosshead speed (v = 10^3 mm/min), the fracture toughness value increased sharply up to the brittle-ductile transition temperature (115^{o}C) then dropped off. The glass transition temperature was 144^{o}C. This reduction can be explained by a reduced molecular mobility at higher loading rate.

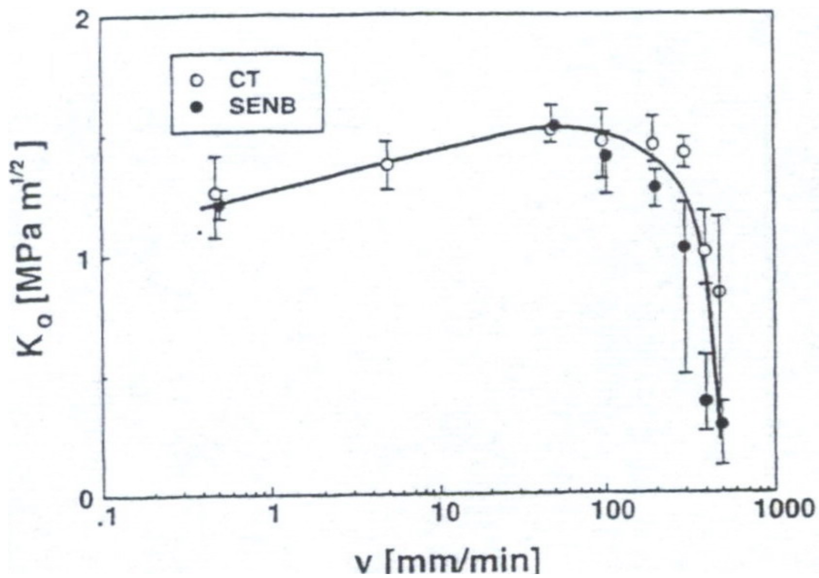


Figure 2.5 Fracture toughness of CT and SENB specimens of TPX at different crosshead rates at ambient temperature [33].

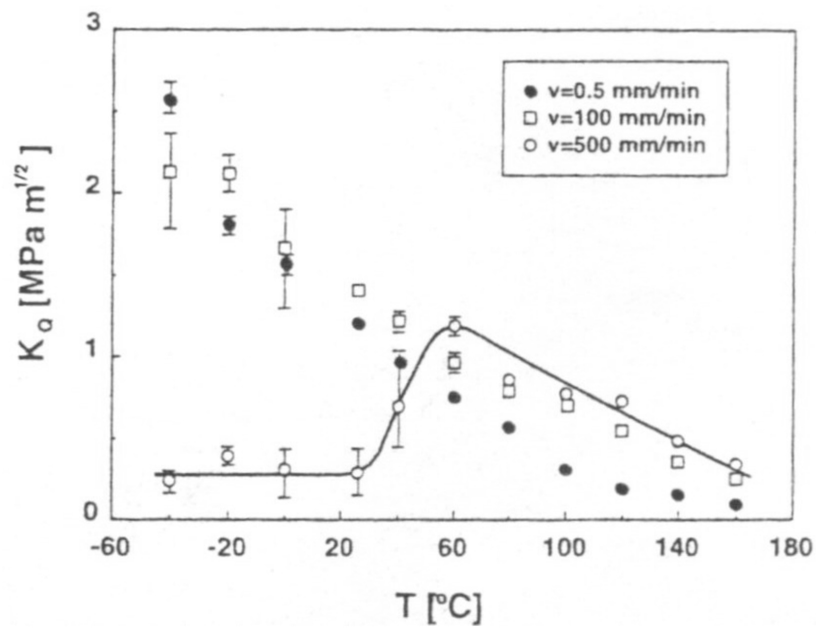


Figure 2.6 Effects of temperature on fracture toughness of TPX at three different crosshead rates [33].

Han et al. [14] observed a strong strain rate and temperature dependence of fracture toughness of phenolphthalein polyether ketone (PEK-C). They found that the fracture toughness increased with increasing temperature and peaked at 160^{o}C then dropped off with further increasing temperature. T_g was 215^{o}C. The fracture toughness decreased with increasing strain rate at a constant temperature.

Percorini and Hertzberg [40] investigated the fracture toughness of annealed poly (ethylene terephthalate) (PET). Their tests were performed on notched three-point bend samples loaded in displacement control at a rate of 10 mm/min. Fracture toughness values varied greatly depending upon the associated annealing treatment. Annealing PET at a Hoffman regime III crystallization temperature (120^{o}C) or inducing solid-state polymerization resulted in materials of high fracture toughness (K_c = 8.7 MPa.m$^{1/2}$ and 9.5 MPa.m$^{1/2}$, respectively). In addition, annealing fully dried PET at 200^{o}C transformed the material to a Hoffman regime I/II structure and the fracture toughness decreased to 6.5 MPa.m$^{1/2}$.

Kim and Ye [23] studied the fracture behavior of polyetherimide (PEI) thermoplastic polymer using compact tension (CT) specimens with special emphasis on effects of specimen thickness and testing temperatures on the plane strain fracture toughness. The results showed that the fracture toughness value remained almost constant with a value of 3.5 MPa.m$^{1/2}$ in the temperature range of 25 to 130^{o}C.

Marshal et al. [41] reported the fracture toughness as a function of crosshead rate for Polystyrene (PS) at 20^{o}C at the crosshead rate range of 10^{-3} to 10^{2} cm/min using SEN specimens. The rate dependence of fracture toughness was very slight except above 10 cm/min where there was a sharp rise. In another study, Parvin and Williams [42] showed the temperature dependence of fracture toughness for PS in the temperature range of -180 to 100^{o}C. The fracture toughness increased up to 0^{o}C then decreased sharply.

Fernando and Williams [24] studied the variation of fracture toughness with temperature for the modified Polypropylene (PP) using SENB in the temperature range of -170 to -30^{o}C. Below -100^{o}C, the fracture toughness remained almost constant, but between -100 and -30^{o}C there was a slight increase in fracture toughness.

Chan and Williams [25] observed the effect of temperature on fracture toughness for Polyethylene (PE) in the temperature range of -180 to 23^{o}C. The fracture toughness decreased in the range of -180 to -120^{o}C thereafter remained fairly constant.

Nimmer et al. [43] reported the temperature dependence of the apparent fracture toughness of a rubber-toughened thermoplastic material as measured using compact-tension specimens in the temperature range of -40 to 30^{o}C. Below -20^{o}C, the fracture toughness increased with temperature. In the vicinity of -20^{o}C, there was a very sudden increase in the values of fracture toughness. After -10^{o}C, the fracture toughness remained constant.

Irfan-ul-Haq and Merah [44] studied the effect of temperature on apparent fracture toughness, maximum value of effective stress intensity factor before failure, of CPVC pipe fittings material at temperatures of -16, 0, 23, 50 and 70^{o}C. They found that the apparent fracture toughness increased with increasing temperature. Also, they found that the toughness, area under stress-strain curve up to the point of fracture, increased with increasing temperature.

Merah et al. [45] investigated the effect of strain rate and temperature on fracture toughness of CPVC pipe fitting material at temperatures of -16, 0, 23 and 50^{o}C and crosshead displacement rates, v, of 5, 50 and 500 mm/min. The fracture toughness tests were performed using a single-edge-notch bending specimens. They found that the effect of crosshead displacement rate on the fracture toughness was insignificant at low temperatures. The value of fracture toughness was calculated using the approximate relationship between the *CTOD* and the displacement of the piston. The fracture toughness was found to remain practically unchanged with an average of 4 MPa.m$^{1/2}$ in the -16 to 23^{o}C temperature range dropping sharply at 50^{o}C.

From the literature review, it can be concluded that the effect of temperature on tensile properties, yield stress, elastic modulus and fracture strain, of CPVC has been studied by few researchers. They found that the yield stress and elastic modulus decreased linearly with increasing temperature and fracture strain increased with temperature. However, there is no work reported on the effect of strain rate on the mechanical properties of CPVC pipe material.

It is seen that no work was reported on the effects of strain rate and temperature on the measured value of fracture toughness of CPVC in the open literature, but other polymers, such as PVC, PC and PMMA, which are closely related to CPVC have been studied by several researchers. They found that the fracture toughness generally increased with temperature and usually decreased with higher strain rate.

On the basis of the literature survey, it can be concluded that the effect of strain rate and temperature on tensile properties and fracture toughness of CPVC is an open area for research and the results will be a useful addition to the existing literature. With knowledge of the relationships between these factors, design engineers can be better equipped to anticipate and thus prevent structural failures.

CHAPTER 3

EXPERIMENTAL PROCEDURE

The main objective of this study was to investigate the effects of strain rate and temperature on fracture toughness and tensile properties of CPVC. This chapter will explain the experimental procedure used in this study. Included in this discussion is the specimen preparation for fracture toughness and tensile tests. The testing equipment and instrumentation will be presented along with the testing program.

3.1 Specimen Preparation

The specimens for fracture toughness and tensile testing programs were prepared from commercially available four-inch CPVC schedule 80 (wall thickness 9.5 mm) pipes. Fifty millimeters wide rings were cut from the pipes and slit for flattening as shown in figure 3.1. After heating for 60 minutes at $120^{o}C$ in an electric oven, the rings were straightened in a specially designed mold (figure 3.2).

The specimens for fracture toughness and tensile tests were then machined from the straightened plates. Coolants were used during machining processes to avoid any localized thermal degradation of material from the heat generated during machining. The fracture toughness specimen for this study was the single edged notched three point bend type. The specimens were produced with the dimensions of $19x84x9.5\ mm^3$ as per

ASTM D5045-93 Standard test methods for plane strain fracture toughness of plastic materials [46]. An initially sharp flaw 1 mm deep was made by pressing a razor-notch guillotine at the end of the machined slot. Figure 3.3 is a photograph of the specially designed and built fixture for the razor-notch guillotine. To ensure a sharp notch tip, a fresh single-edged stainless steel razor blade was used for each specimen. Figure 3.4 shows the geometry and dimensions of fracture toughness specimen.

For tensile tests, type III specimens were prepared from the rigid plates with a thickness of 9.5 mm according to the ASTM D638-01 Standard method of test for tensile properties of plastics [47]. The geometry and dimensions of the tensile specimen used in the present testing program are illustrated in figure 3.5.

3.2 Testing Apparatus

The following section details the testing apparatus used in the present study. Included is a description of Instron 8801 material testing machine, clip gauge (Instron 2670-116), environmental chambers, video recording equipment, digital camera system (Pixera) and scanning electron microscope (SEM).

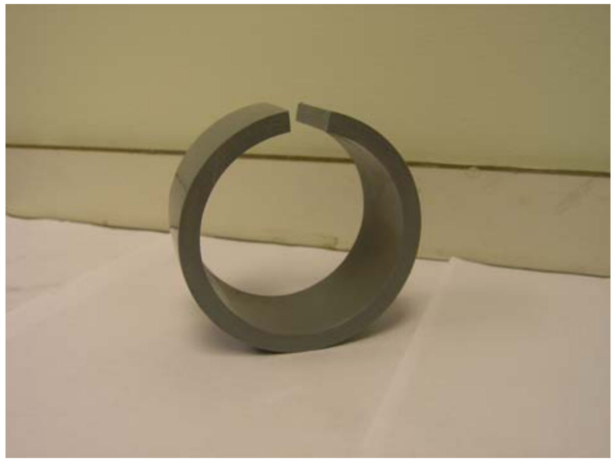


Figure 3.1 Ring of width equal to 50 mm were cut from CPVC pipe.

Figure 3.2 Specially designed mold for straightening Specimens.

Figure 3.3 Photograph of the razor-notch guillotine and SENB specimen.

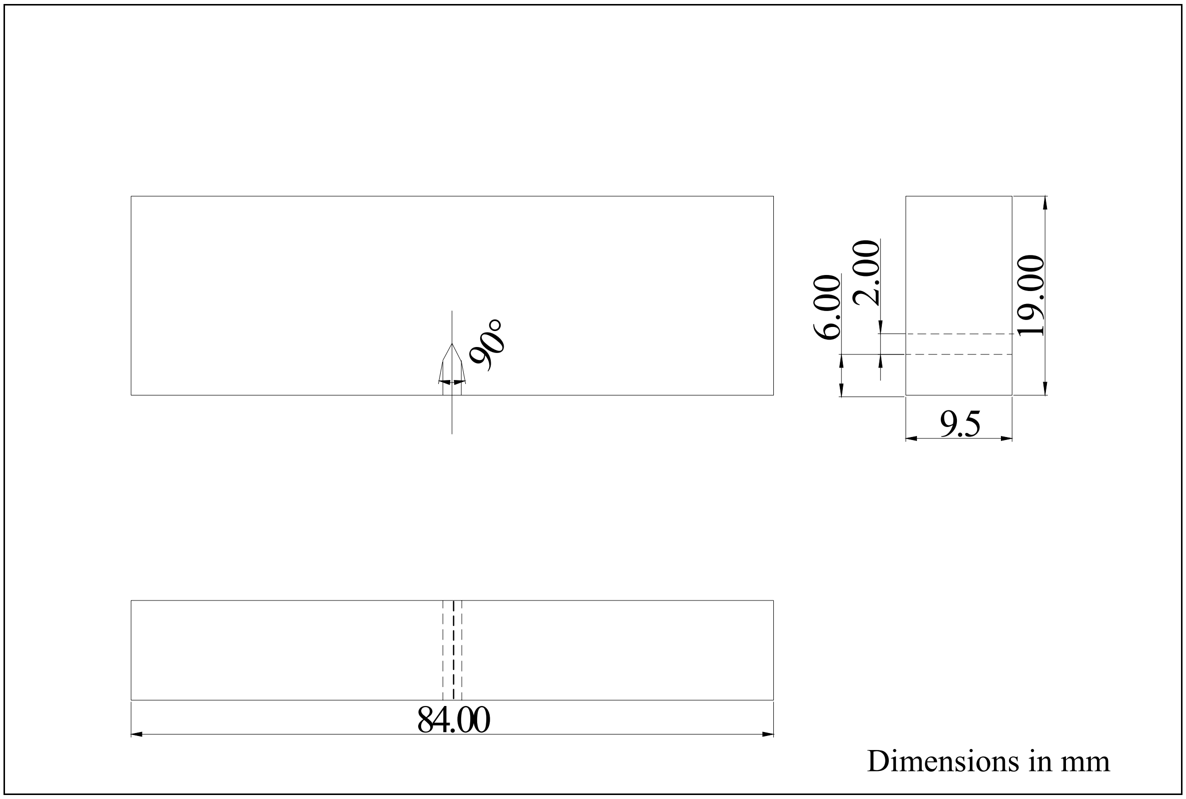


Figure 3.4 Geometry and dimensions of the SENB specimen used in this study.

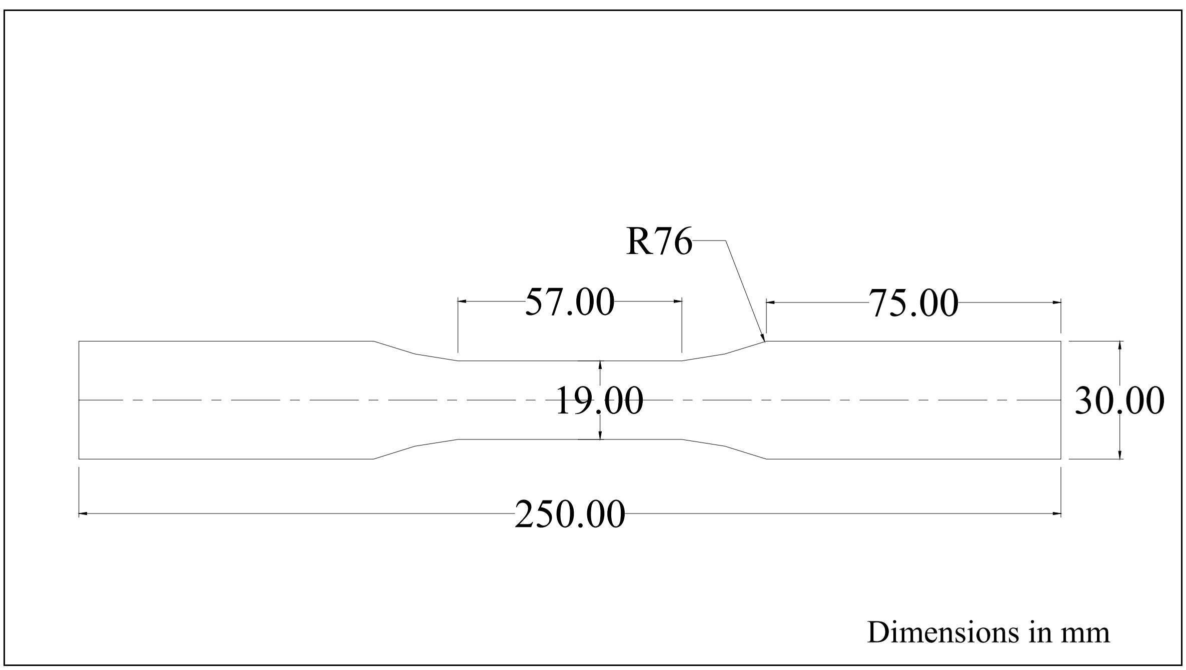


Figure 3.5 Geometry and dimensions of the tensile specimen used in the study.

3.2.1 Instron 8801

The Instron 8801 machine was used to perform both fracture toughness and tensile tests. It is a closed loop servo-hydraulic single axis fatigue testing system. The main controlling modes of the system are strain (±10 %), load (±100 kN) and position (±75 mm) with a frequency range from 0 to 200 Hz. The controlling limits can be viewed on the digital control panel any time during the test along with all other test variables. The photograph of testing frame with video camera and environmental chamber is shown in figure 3.6.

The machine is equipped with a hydraulically actuated self-aligning gripping system. To ensure the vertical alignment of the specimen specially machined inserts were used during the tests. Any pre-loading induced during clamping was adjusted to zero prior to testing by the re-calibration of the load cell after clamping.

The fracture toughness and tensile tests were carried out in position control mode. The Instron BlueHill Series machine control data acquisition and analysis software for material testing will be used. A PC, interfaced with the testing frame was used for test data acquisition. The software provided position and corresponding load of the test with a constant position increment till fracture at the ultimate tensile strength which was logged along with the final position before fracture.

Figure 3.6 Photograph of testing frame with video camera and environmental chamber.

3.2.2 Clip Gauge (Instron 2670-116)

The clip gauge, Instron crack opening displacement (COD), is the most common displacement transducer in fracture toughness tests (figure 3.7). It was attached to two sharp knife-edges which were glued to the specimen at the crack mouth as shown in figure 3.8. The COD gauge gave a precise indication of the relative displacement of accurately located knife-edges which spanned the starter notch of the specimen and it provided a linear output signal proportional to displacement. The clip gauge consists of four resistance strain gages bonded to a pair of cantilever beams. Deflection of the beams resulted in a change in voltage across the strain gages, which varied linearly with crack opening displacement. The specifications of Instron COD gauge are gauge length (10 mm), maximum travel (4 mm), linearity error (±0.1% of reading), frequency range (up to 50Hz) and operating temperature (-20 to 60^{o}C).

3.2.3 Environmental Chambers

Four environmental chambers designed and fabricated by Irfan-ul-Haq [6] were modified for conducting fracture toughness and tensile tests in non-ambient environments. Two high temperature environmental chambers, one for fracture toughness test and the other for tensile test, were designed to maintain temperatures up to 150^{o}C for extended periods of time. They were made from Plexiglas and insulated with glass wool embedded in aluminum foil. Two 10 x 2 inch electric heating tapes were used for heating the chambers. The temperature within the chamber was maintained within ±0.50^{o}C from the target temperature. An automatic feed back system consisting of a self-adhesive J-type thermocouple (maximum temperature range of 177^{o}C) and an electronic controller

Figure 3.7 Photograph of Instron gauge 2670-116.

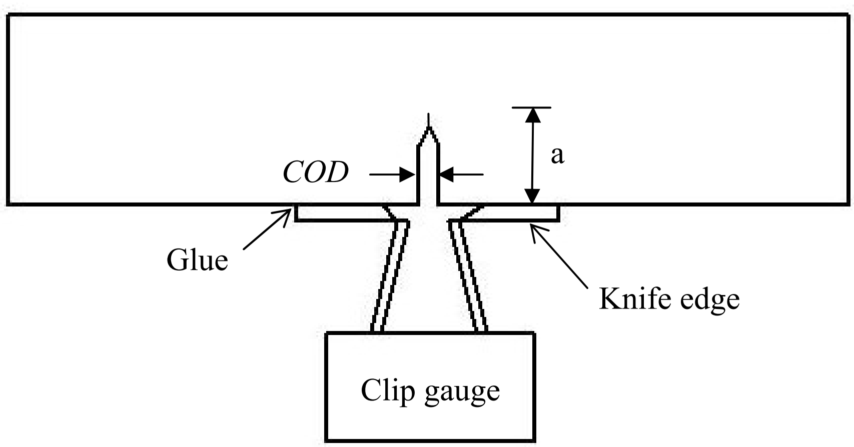


Figure 3.8 Measurement of the crack opening displacement with a clip gauge.

were employed for controlling the temperature. The chambers were calibrated by operating them for 24 hours.

The other chambers for low temperature fracture toughness and tensile tests were also made from Plexiglas and insulated with rubber sheet embedded in aluminum foil. The chamber is cooled by circulating the cooling fluid through a copper coil (10mm diameter). The cooling fluid used was a mixture of 30% water and 70% ethylene glycol. A cold fluid reservoir and a circulating pump were used to maintain the flow of fluid through the copper coil. Two small circulating fans were employed to ensure uniform temperature distribution inside the chamber. A J-type thermocouple was attached to the specimen and the temperature of the specimen was read on a digital thermometer. This temperature was used as the feed back for manual control of temperature in the chamber. The temperature in the chamber was controlled by varying the fluid temperature or fluid flow rate. The low temperature environmental chambers were also calibrated for 24 hours before actual tests were started. In each of these chambers a window (30x60 mm^2) was designed to allow video recording of specimens during testing. The cooling environmental chamber employed in fracture toughness test is shown in figure 3.9.

3.2.4 Digital Camera System

The fracture surface morphology of the failed specimens was studied using a digital camera system (Pixera), as shown in figure 3.10. The Pixera Professional is a versatile, 1.2 million pixel color digital camera system that provides excellent image quality (magnification 28x), color reproduction, sensitivity (2.5 lux) and resolution (1260

Figure 3.9 Photograph of cooling environmental chamber for fracture toughness test.

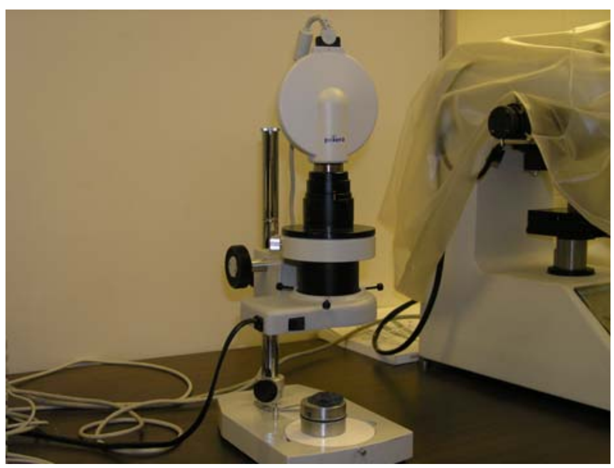


Figure 3.10 Photograph of digital camera system (Pixera).

x 960). The analysis of fracture surfaces is performed by utilizing Pixera's unique optical DiRactor™ (Diafram Refractor) and software image processing technology.

3.2.5 Scanning Electron Microscope

The fractography of the fractured surfaces was studied using a Joel JSM scanning electron microscope (SEM). The magnification range available is 10x to 300,000x. The excitation potential can be varied between 1 to 50 kV. To suppress charging and increasing electron emission, gold coating of the fracture surface was done. This process provided very fine uniform coating of a conducting material (i.e. gold), so that the surface after coating was an exact replica of the underlying material. The coating was done by vacuum depositing in several stages provided by rotary and diffusion pumps of Joel Fine Coat Ion Sputter JFC-1100 with a vacuum of 0.1 Torr at 1-1.5 kV. The time and amperage were selected with the constraint of the coat thickness and the specimen surface area.

3.3 Testing Program

The investigation of the effects of strain rate and temperature on fracture toughness and tensile properties of CPVC is the main objective for this study. This section will explain the testing program used in the present study, including the tensile tests and fracture toughness at different crosshead speeds and temperatures. The results obtained from these tests will be analyzed and discussed in the following chapters.

3.3.1 Tensile Test

Tensile tests were conducted at each of these temperatures: -10, 0, 23, 50 and 70^{o} C and at crosshead speeds of 5, 50 and 500 mm/min. According to the ASTM D638-01 Standard, two to three tests were conducted at each condition as shown in Table 3.1. Additional tests at crosshead speeds of 100 and 300 mm/min were performed at room temperature. The data obtained from tensile tests provide information on the yield stress, elastic modulus and fracture strain as well as toughness modulus. This information is used in the present study to investigate the effect of strain rate and temperature on tensile properties of CPVC.

3.3.2 Fracture Toughness Test

The fracture toughness tests on CPVC single edge notch bending specimens, described in section 3.1, were conducted at -10, 0, 23, 50 and 70^{o}C for three crosshead displacement rates 5, 50 and 500 mm/min. According to ASTM D5045-93 Standard, three tests were run at each condition as shown in Table 3.2. At each test, the load P and crack opening displacement *COD* were recorded using a PC interfaced with the testing frame and video camera system.

Table 3.1 Test matrix for tensile tests of CPVC specimens.

Temp. / v(mm/min)	-10ºC	0ºC	23ºC	50ºC	70ºC
5	2	2	2	3	3
50	2	2	2	3	3
500	2	2	2	3	3

Table 3.2 Test matrix for fracture toughness measurement of CPVC specimens.

Temp. / v(mm/min)	-10ºC	0ºC	23ºC	50ºC	70ºC
5	3	3	3	3	3
50	3	3	3	3	3
500	3	3	3	3	3

CHAPTER 4

EFFECTS OF STRAIN RATE AND TEMPERATURE ON TENSILE PROPERTIES

In this chapter, the results of tensile tests obtained at different crosshead speeds and temperatures will be presented. The effects of strain rate (crosshead speed) and temperature on the tensile properties such as yield stress, modulus of elasticity and fracture strain will be then discussed.

4.1 Results

In the present study, the tensile tests were conducted on CPVC pipe material specimens at -10, 0, 23, 50 and 70^{o}C and crosshead speeds of 5, 50 and 500 mm/min. The average values of tensile properties such as yield stress, elastic modulus and fracture strain are summarized in Tables 4.1 through 4.5. According to the definition in ASTM D638-01, the yield stress is regarded as the tensile stress (maximum stress) at yield and the elastic modulus is the ratio of stress to strain when deformation is totally elastic. In addition, the fracture strain is defined as the strain at which specimens break.

The yield stress values for this CPVC material are lower than those reported in other research work [6, 34] whereas the elastic modulus and fracture strain values are comparable. It is observed that the stress-strain properties of CPVC are sensitive to the strain rate and temperature. Figures 4.1, 4.2 and 4.3 show the engineering stress-strain curves obtained from the load-elongation data using a PC interfaced with the testing frame. Several features of these curves are worth noting; increasing the temperature produces a decrease in elastic modulus, a reduction in tensile strength, an increase in fracture strain thus an enhancement of ductility. At -10 and 0^{o}C, there is a limited amount of plastic deformation, whereas considerable plastic deformation is realized at 23, 50 and 70^{o}C for all crosshead speeds. In general, decreasing the strain rate (crosshead speed) has the same influence on tensile properties as increasing the temperature and the material becomes softer and more ductile. The complete analysis of the effect of strain rate and temperature on yield stress, elastic modulus and fracture strain will be presented in sections 4.2, 4.3 and 4.4, respectively.

4.2 Yield Stress

A normal tensile test on a polymer produces a stress-strain curve similar to that of a metal. As the strain is increased, the material passes through a recoverable elastic region, which in contrast to metals is usually non-linear. The slope of the curve decreases until it reaches a peak value in stress, which can be used to define the yield stress, σ_{ys}. Strictly speaking, the yield point of the material should be described as the point at which permanent set takes place. This is very difficult to define in certain polymers because there is no clear distinction between elastic recoverable deformation and plastic irrecoverable deformation [10].

Table 4.1 Results of tensile tests performed at -10^{o}C for different crosshead speeds.

Serial No.	Yield Stress (MPa)	Elastic Modulus (MPa)	Fracture Strain (%)
5 mm/min			
1	59.17	3398.12	8.38
2	59.19	3465.64	6.41
Average	**59.18**	**3431.88**	**7.40**
50 mm/min			
3	64.92	3620.00	7.29
4	65.37	3520.00	6.25
Average	**65.15**	**3570.00**	**6.77**
500 mm/min			
5	73.58	3688.78	6.18
6	72.97	3814.36	7.12
Average	**73.28**	**3751.57**	**6.65**

Table 4.2 Results of tensile tests performed at $0^{o}C$ for different crosshead speeds.

Serial No.	Yield Stress (MPa)	Elastic Modulus (MPa)	Fracture Strain (%)
5 mm/min			
1	56.03	3179.27	5.73
2	55.13	3160.27	7.03
Average	**55.58**	**3169.77**	**6.38**
50 mm/min			
3	65.03	3580.00	4.68
4	63.77	3520.00	5.73
Average	**64.40**	**3550.00**	**5.21**
500 mm/min			
5	71.16	3548.44	7.96
6	71.68	3648.59	7.07
Average	**71.42**	**3598.52**	**7.52**

Table 4.3 Results of tensile tests performed at 23°C for different crosshead speeds.

Serial No.	Yield Stress (MPa)	Elastic Modulus (MPa)	Fracture Strain (%)
5 mm/min			
1	47.17	2876.07	16.84
2	45.82	2890.10	8.12
Average	**46.50**	**2883.09**	**12.48**
50 mm/min			
3	53.50	3073.73	13.54
4	54.51	2737.44	14.08
Average	**54.01**	**2905.59**	**13.81**
100 mm/min			
5	54.57	2965.01	15.63
6	55.20	2906.71	15.36
Average	**54.89**	**2935.86**	**15.50**
300 mm/min			
7	58.30	3042.95	16.77
8	55.67	3199.53	11.88
Average	**56.99**	**3121.24**	**14.33**
500 mm/min			
9	61.26	3291.49	10.72
10	62.17	3181.27	13.12
Average	**61.72**	**3236.38**	**11.92**

Table 4.4 Results of tensile tests performed at 50°C for different crosshead speeds.

Serial No.	Yield Stress (MPa)	Elastic Modulus (MPa)	Fracture Strain (%)
5 mm/min			
1	34.61	2682.36	10.62
2	34.26	2628.13	8.36
3	34.37	2458.61	14.06
Average	**34.41**	**2589.70**	**11.01**
50 mm/min			
4	35.15	2680.00	17.50
5	38.56	2680.00	13.33
6	36.80	2600.00	16.46
Average	**36.84**	**2653.33**	**15.76**
500 mm/min			
7	36.78	2625.65	18.69
8	44.71	2861.13	18.23
9	41.82	2791.60	16.40
Average	**41.10**	**2759.46**	**17.77**

Table 4.5 Results of tensile tests performed at 70°C for different crosshead speeds.

Serial No.	Yield Stress (MPa)	Elastic Modulus (MPa)	Fracture Strain (%)
5 mm/min			
1	30.07	2062.70	14.11
2	29.84	2016.78	12.37
3	28.60	2361.54	14.68
Average	**29.50**	**2147.01**	**13.72**
50 mm/min			
4	32.21	2120.00	40.10
5	31.33	2120.00	34.58
6	33.73	2200.00	37.49
Average	**32.42**	**2146.67**	**37.39**
500 mm/min			
7	37.56	2268.39	38.99
8	36.89	2330.23	33.43
9	35.26	2437.20	33.90
Average	**36.57**	**2345.27**	**35.44**

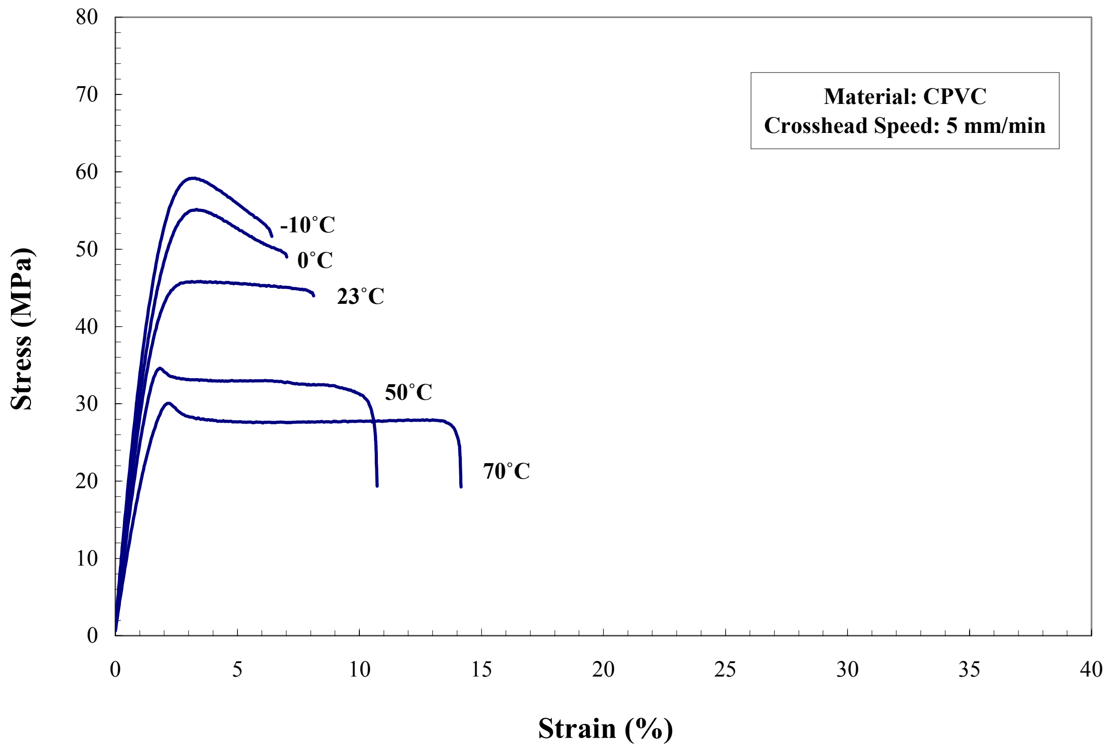


Figure 4.1 Stress-strain curves for CPVC at 5 mm/min for different temperatures.

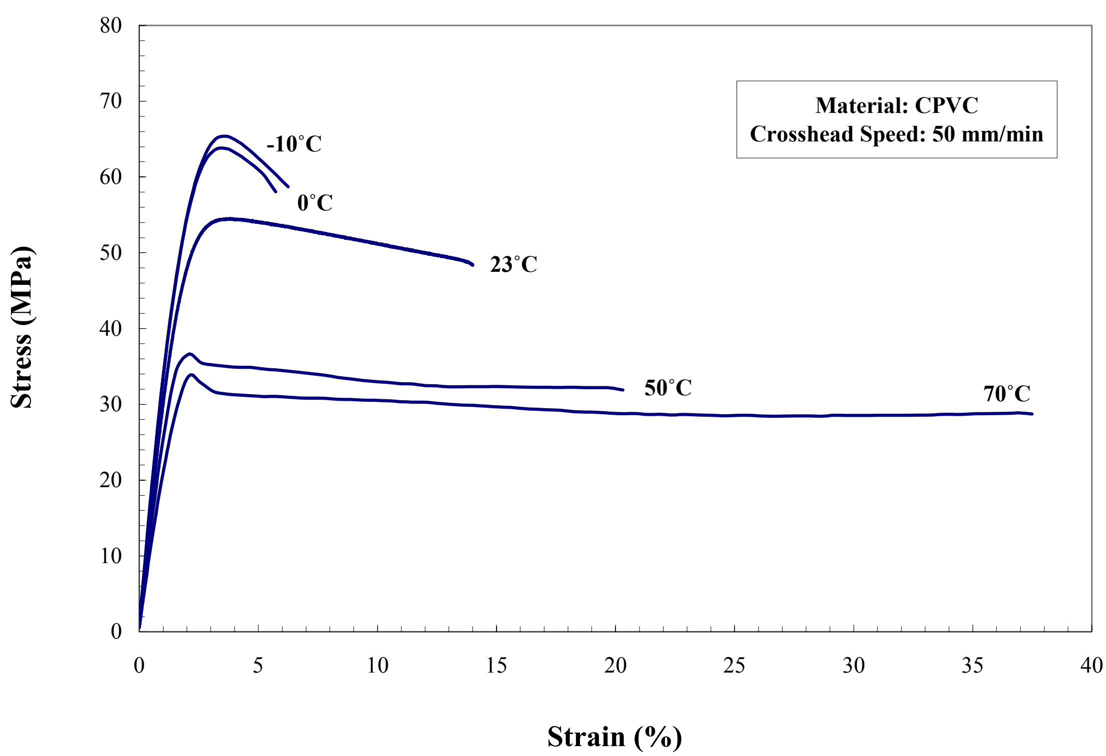


Figure 4.2 Stress-strain curves for CPVC at 50 mm/min for different temperatures.

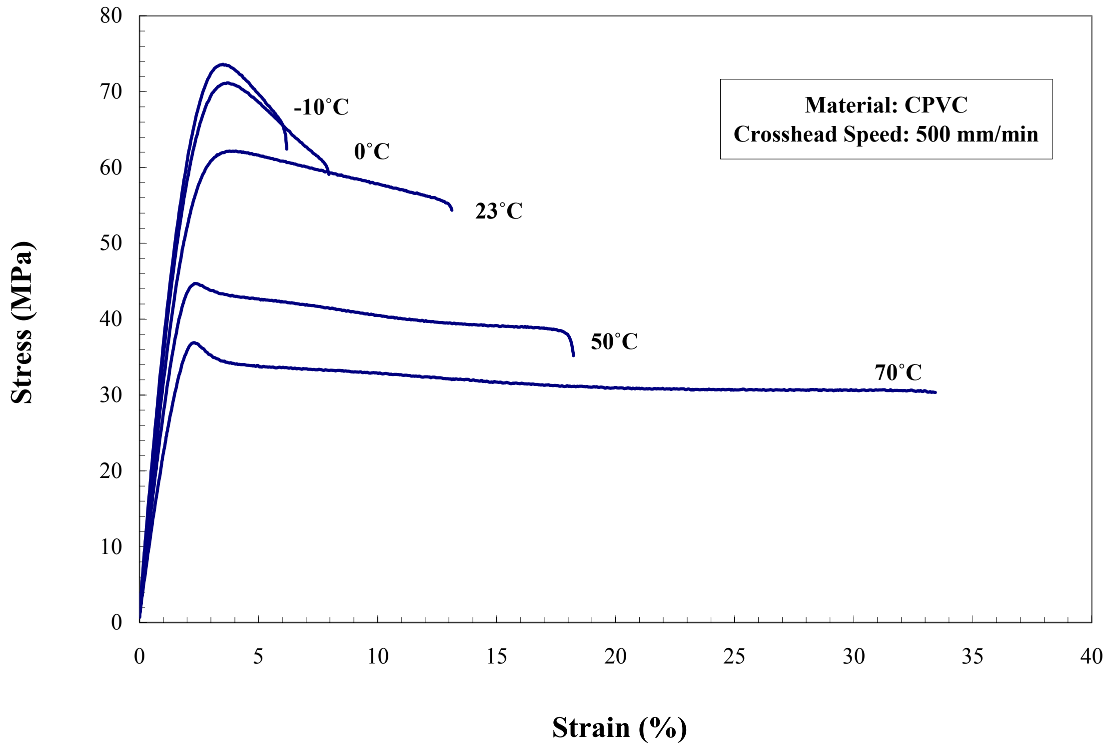


Figure 4.3 Stress-strain curves for CPVC at 500 mm/min for different temperatures.

In this section, the effect of strain rate (crosshead speed) and temperature on yield stress is investigated. The qualitative descriptions of the combined effects of these two parameters are displayed in figure 4.4. It can be seen that the tensile yield stress increases with crosshead speed at all temperatures. However, it decreases with increasing temperature at all crosshead speeds. The maximum value of yield stress occurs at -10^{o}C and 500 mm/min whereas the minimum value occurs at 70^{o}C and 5 mm/min.

4.2.1 Effect of Strain Rate on Yield Stress

The strain rate (crosshead speed) dependence of yield stress for most polymeric materials has been investigated and reported in the literature. In general, the tensile yield stress for PVC increased with increasing crosshead speed.

The yield stress as a function of crosshead speed at the temperatures of -10, 0, 23, 50 and 70^{o}C for CPVC is shown in figure 4.5. It is observed that the yield stress increases generally as the crosshead speed increases. The enhancement in yield stress can be explained by a reduced molecular mobility due to short time available for plastic zone to form or an increased in strain hardening at high crosshead speed and thus a low ductility is expected.

The variation of yield stress with crosshead speed can be expressed as:

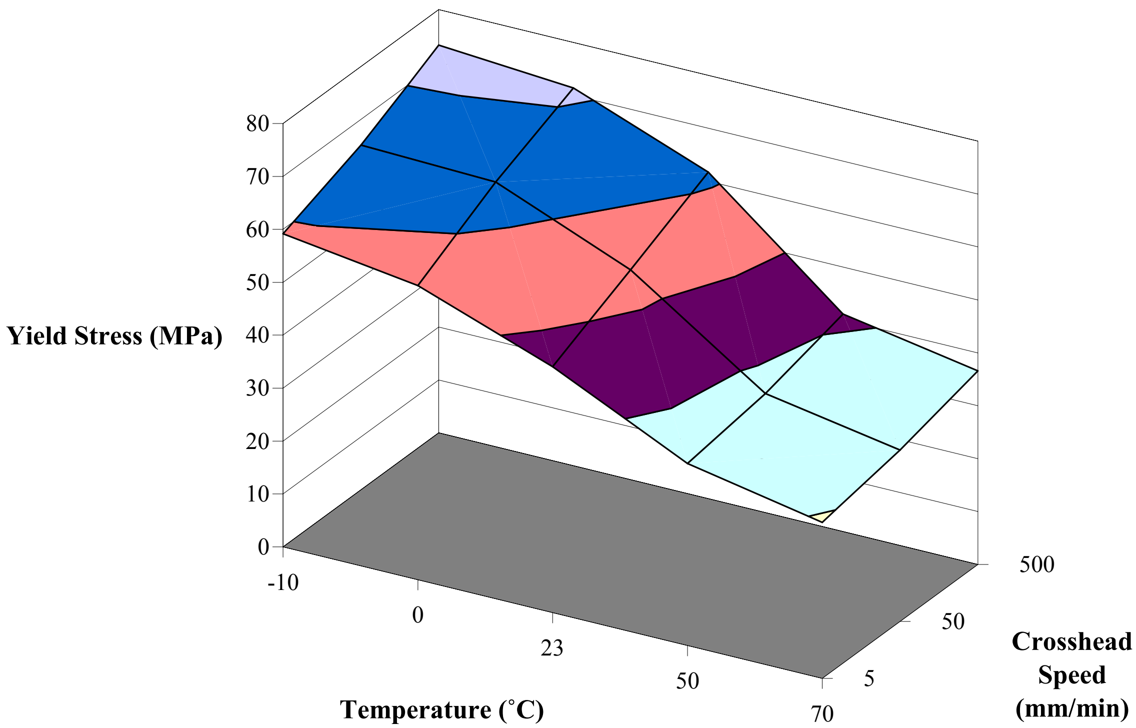


Figure 4.4 Crosshead speed and temperature dependence of yield stress for CPVC (not to scale).

$$\sigma_{ys} = 53.89 + 7.05 \log (v) \qquad \text{for } T = -10^{o}C \qquad (4.1)$$

$$\sigma_{ys} = 50.34 + 7.92 \log (v) \qquad \text{for } T = 0^{o}C \qquad (4.2)$$

$$\sigma_{ys} = 41.66 + 6.86 \log (v) \qquad \text{for } T = 23^{o}C \qquad (4.3)$$

$$\sigma_{ys} = 31.77 + 3.34 \log (v) \qquad \text{for } T = 50^{o}C \qquad (4.4)$$

$$\sigma_{ys} = 26.82 + 3.55 \log (v) \qquad \text{for } T = 70^{o}C \qquad (4.5)$$

Crosshead speed has more effect on yield stress at lower temperatures ($T \leq 23^{o}C$). The slope of the line at 50 and 70^{o}C is about half of that at lower temperatures. At these temperatures, the failure is mainly ductile and depends less on the strain rate. In general, the yield stress decreases with increasing temperature. However, the slope seems to remain constant at about 7.0 between -10 and 23^{o}C and at about 3.4 between 50 and 70^{o}C. This is indicative of two operative mechanisms brittle (shear controlled) failure in the low temperature range and ductile (craze controlled) in the high temperature range.

As shown in figure 4.6, CPVC displays behavior very similar to that of PVC [29] with regard to crosshead speed dependence of yield stress at room temperature and over a range of crosshead speed from 0.01 to 100 mm/min. The slopes of CPVC are higher than PVC [29] between 5 and 100 mm/min. The value of yield stress for this CPVC material is lower than PVC [29] probably because of toughening additives in the present CPVC material.

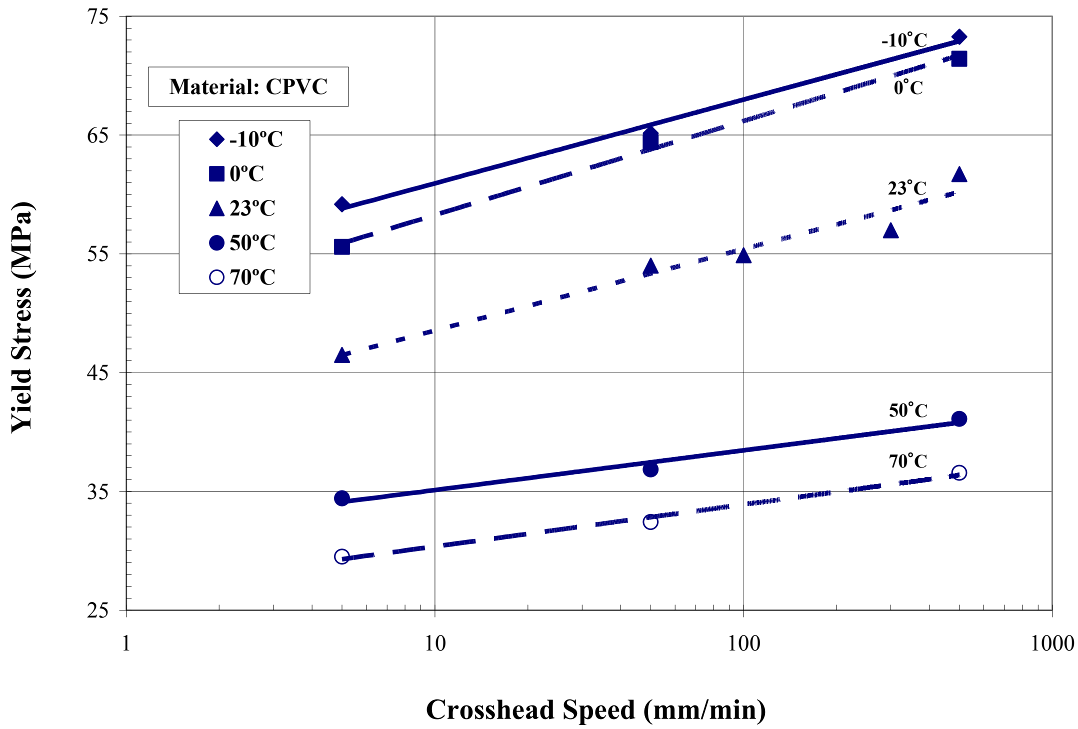


Figure 4.5 Crosshead speed dependence of yield stress for CPVC at different temperatures.

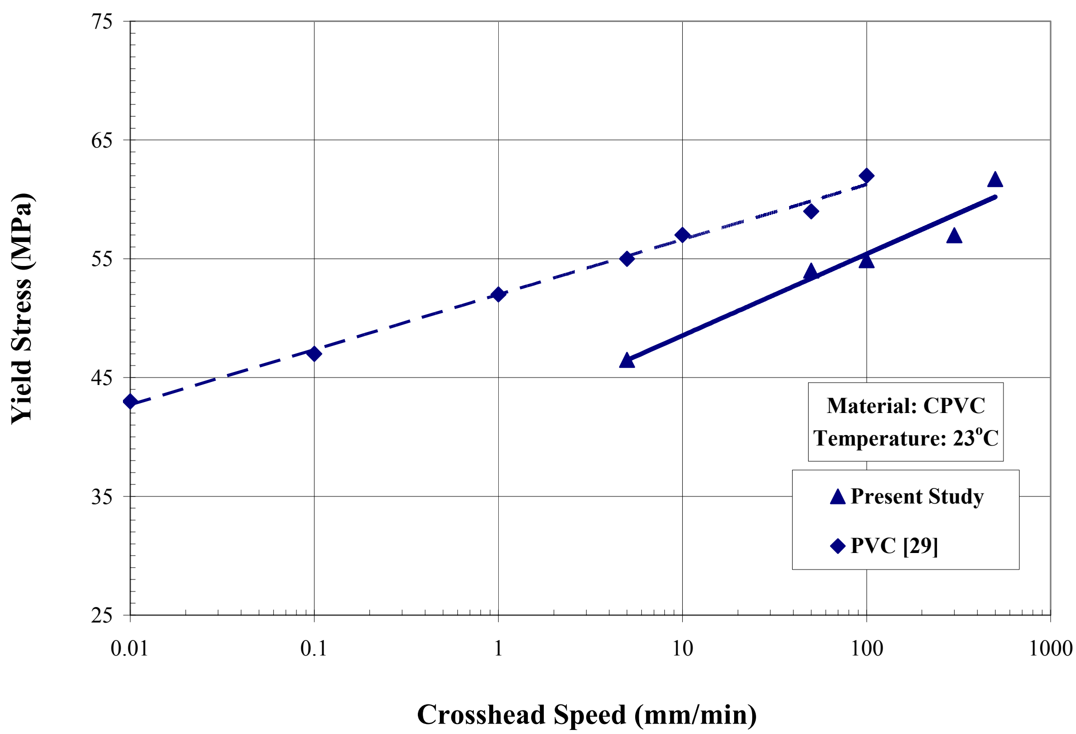


Figure 4.6 Variation of yield stress with crosshead speed at room temperature.

4.2.2 Effect of Temperature on Yield Stress

The temperature dependence of yield stress for different polymers has been studied by many researchers. Merah et al. [35] reported that the yield stress of CPVC material used in pipe fittings decreases with increasing temperature in the temperature range -10 to 70°C at a crosshead speed of 5 mm/min. Similar results were given by other investigators such as Povolo et al. [27] for PVC at a strain rate of $6.6 \times 10^{-4}\ s^{-1}$.

The variation of yield stress with temperature at different crosshead speeds for the present work is shown in figure 4.7. The value of yield stress for different crosshead speeds at -10, 0, 23, 50 and 70°C were given in Tables 4.1 through 4.5. It can be seen that the yield stress decreases linearly with increasing temperature in the temperature range of -10 to 70°C. Even though the entire data can be described by a single line, however, it is clear that yield stress decreases faster between -10 and 50°C than between 50 and 70°C. This reduction can be justified by the positive effect of molecular mobility and plastic deformation.

The linear dependence of yield stress with temperature at different crosshead speeds can be expressed as:

$$\sigma_{ys} = 55.23 - 0.38\, T\,(^{o}C) \qquad \text{for v = 5 mm/min} \qquad (4.6)$$

$$\sigma_{ys} = 62.51 - 0.45\, T\,(^{o}C) \qquad \text{for v = 50 mm/min} \qquad (4.7)$$

$$\sigma_{ys} = 70.27 - 0.48\, T\,(^{o}C) \qquad \text{for v = 500 mm/min} \qquad (4.8)$$

The present results obtained at a crosshead speed of 5 mm/min are compared to those of other CPVC compound [35] at 5 mm/min and PVC [27] at 6.6×10^{-4} s^{-1} in figure 4.8. A rough comparison between the temperature dependence of PVC and CPVC reveals that similar temperature effect on yield stress is observed. In addition, the present study has a lower value of σ_{ys} than CPVC [35] and PVC [27]. This may be attributed to lower quality of this CPVC material. Merah et al. [35] found that the linear dependence of yield strength with temperature at a crosshead speed of 5 mm/min is described by:

$$\sigma_{ys} = 187 - 0.456\, T\,(\mathrm{K}) \qquad \text{for v = 5 mm/min} \qquad (4.9)$$

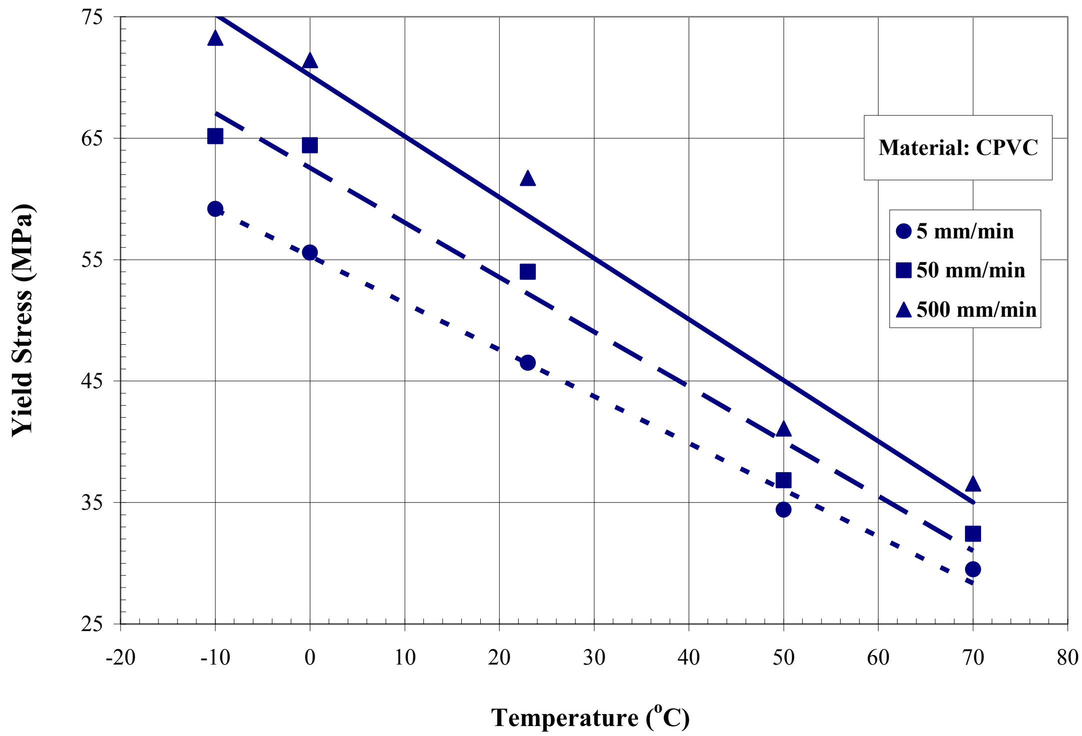


Figure 4.7 Temperature dependence of yield stress for CPVC at different crosshead speeds.

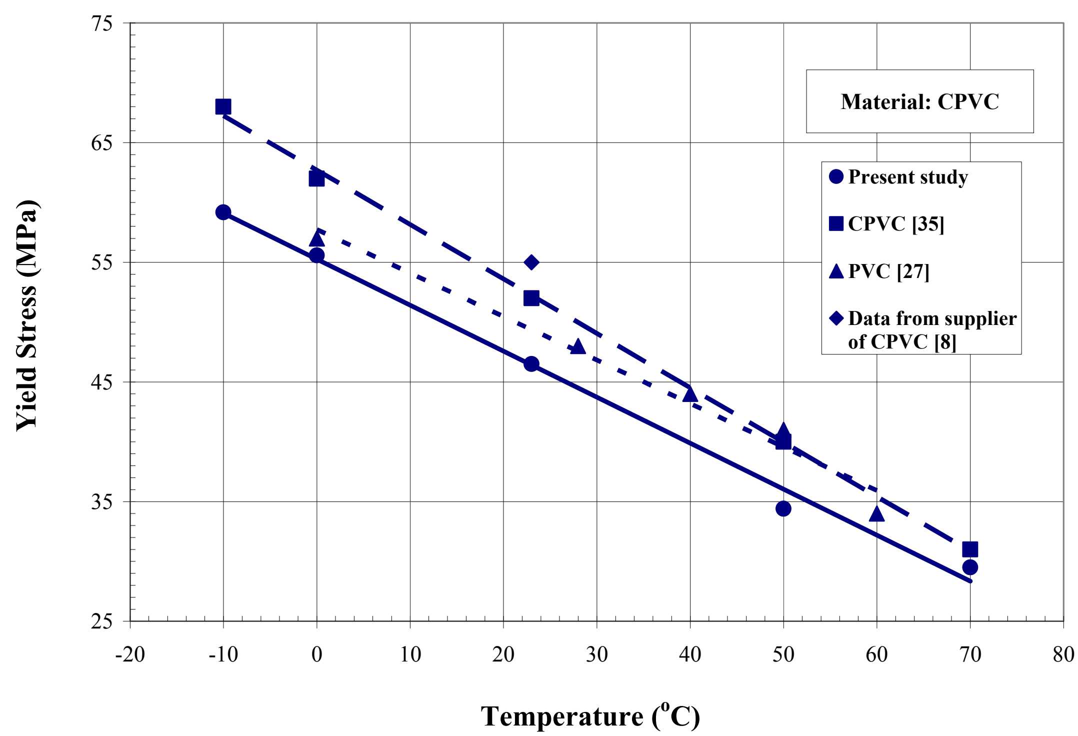


Figure 4.8 Variation of yield stress with temperature at low crosshead speed.

4.3 Modulus of Elasticity

According to Hooke’s law the modulus of elasticity or Young's modulus is defined as:

$$E = \sigma / \varepsilon \tag{4.10}$$

It is the ratio of stress to strain when deformation is totally elastic. It can be also defined as the slope of the tangent to the part of the stress-strain curve which lies in the limit of proportionality. This modulus may be thought of as stiffness, or a material's resistance to elastic deformation. The greater the modulus is the stiffer the material or the smaller the elastic strain that result from the application of a given stress. The modulus is an important design parameter used for computing elastic deformations.

The variation of elastic modulus with crosshead speed and temperature is shown in figure 4.9. It can be seen that the elastic modulus increases with crosshead speed for all temperatures in the crosshead speed range of 5 to 500 mm/min. On the other hand, it decreases with increasing temperature for all crosshead speeds. The material is stiffer at low temperature because of reduced molecular mobility. At high crosshead speed, there is insufficient time for the material to respond to stress with large-scale viscoelastic deformation or yielding. Thus, flexibility is expected in the low temperature-high crosshead speed range. At high temperature, the stiffness of CPVC is low.

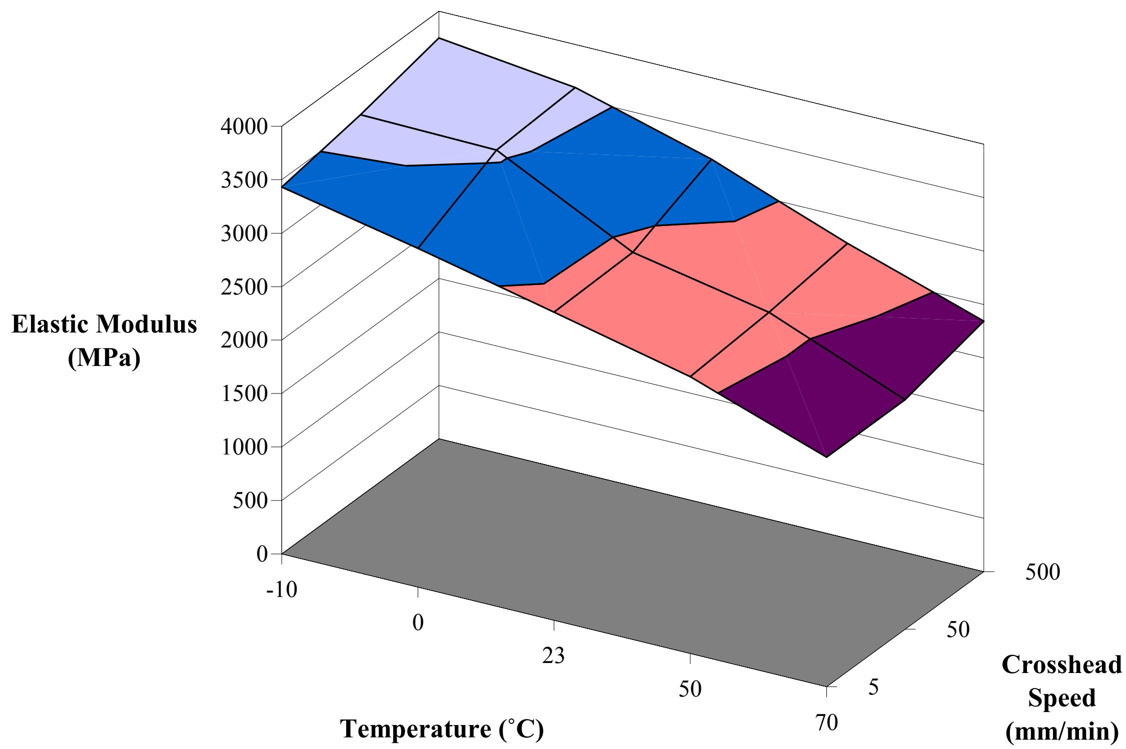


Figure 4.9 Variation of elastic modulus with crosshead speed and temperature (not to scale).

4.3.1 Effect of Strain Rate on Modulus of Elasticity

Strain rate (crosshead speed) has a significant effect on the value of elastic modulus of many polymers. Ye et al. [33] found that the Young's modulus for TPX at ambient temperature increased with crosshead speeds from 0.5 to 500 mm/min. Similar results were reported by Che et al. [29] for PVC.

The variation of elastic modulus of CPVC with crosshead speed obtained in the present study for different temperatures is illustrated in figure 4.10. It can be seen that the elastic modulus or stiffness generally increases with increasing crosshead speed at each test temperature.

The variation of elastic modulus with crosshead speed can be expressed as:

$$E = 3312.9 + 159.85 \log(v) \qquad \text{for } T = -10^{o}\text{C} \qquad (4.11)$$

$$E = 3075.2 + 214.37 \log(v) \qquad \text{for } T = 0^{o}\text{C} \qquad (4.12)$$

$$E = 2697.3 + 166.68 \log(v) \qquad \text{for } T = 23^{o}\text{C} \qquad (4.13)$$

$$E = 2523.3 + 84.87 \log(v) \qquad \text{for } T = 50^{o}\text{C} \qquad (4.14)$$

$$E = 2044.6 + 99.13 \log(v) \qquad \text{for } T = 70^{o}\text{C} \qquad (4.15)$$

The results obtained here at room temperature are compared to those obtained by Che et al. [29] for PVC over the 0.01 to 100 mm/min crosshead speed range in figure 4.11. It can be seen that the crosshead speed has a similar effect on both materials. CPVC seems to be more sensitive to strain rate than PVC. This is indicated by the higher slope. The cause may be attributed to higher chlorine content which makes CPVC more rigid.

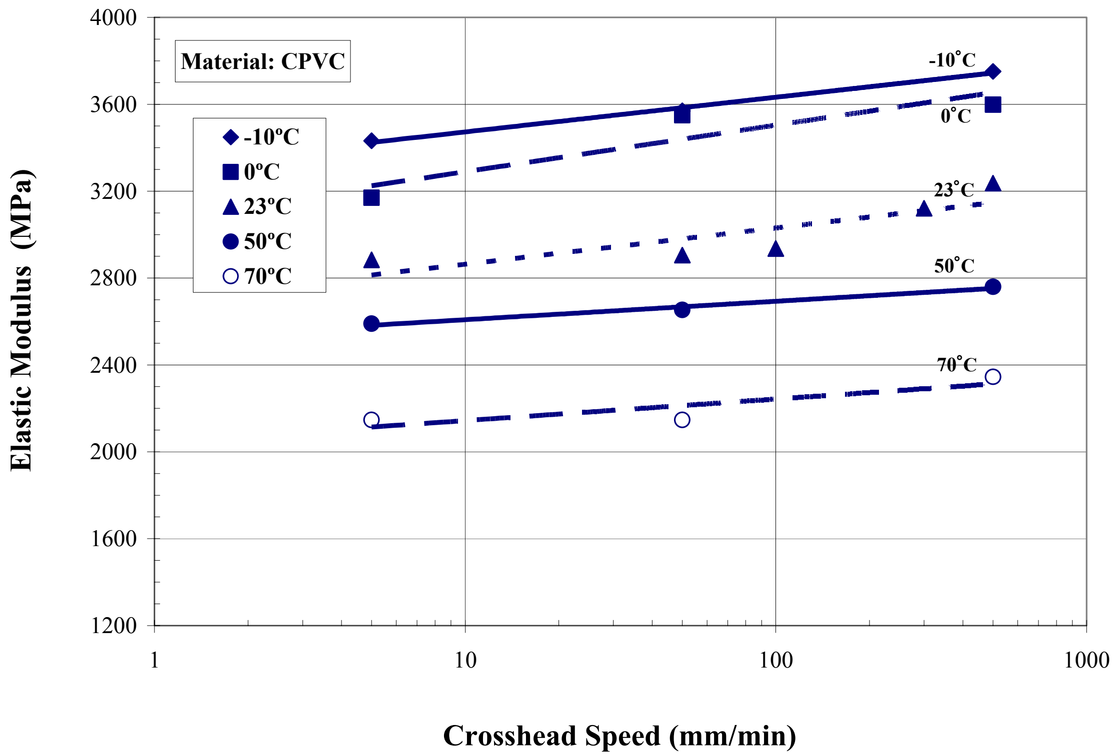


Figure 4.10 Elastic modulus dependence of crosshead speed at different temperatures.

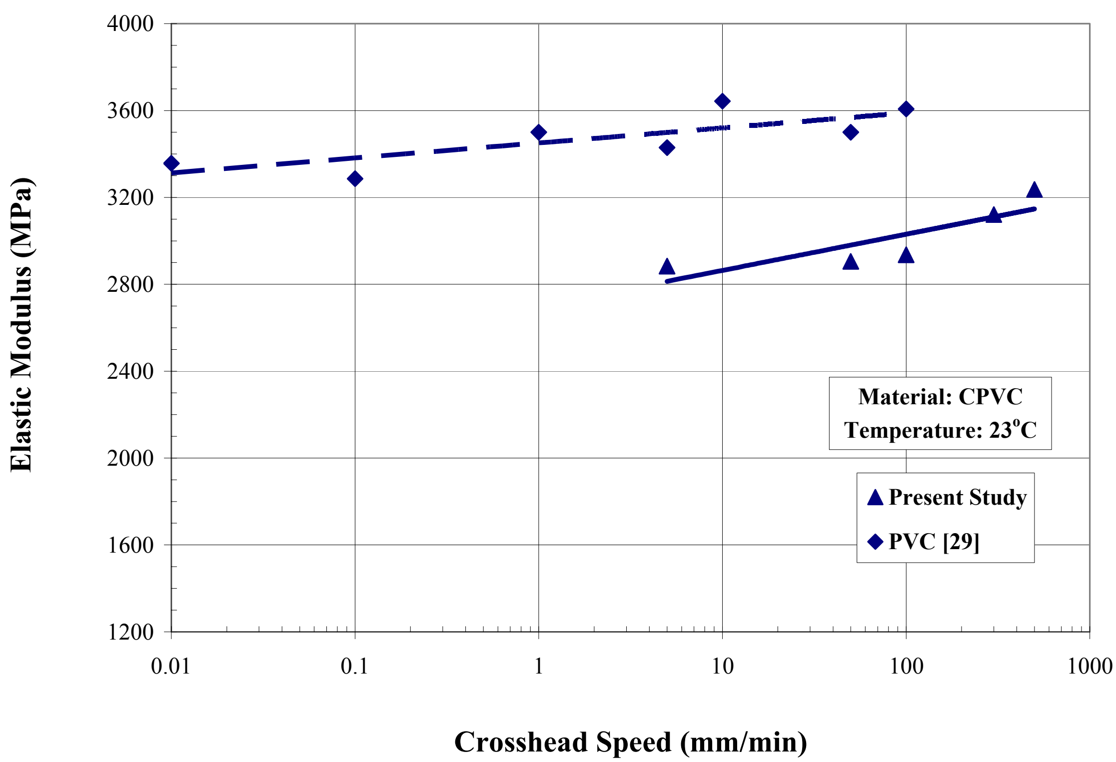


Figure 4.11 Variation of elastic modulus with crosshead speed at room temperature.

4.3.2 Effect of Temperature on Modulus of Elasticity

Figure 4.12 shows the influence of temperature on elastic modulus for CPVC at three different crosshead speeds (5, 50 and 500 mm/min). It can be observed that the elastic modulus or stiffness decreases with increasing temperature over the temperature range of -10 to 70^{o}C. When the temperature increases, thermal activation provides increased free volume between chain molecules permitting motion of increased lengths of chain and consequently the elastic modulus decreases. At 50^{o}C, the elastic modulus decreases rapidly at low and high crosshead speeds. This temperature seems to be near the brittle-to-ductile transition temperature as reported by Irfan-ul-Haq [6] and Saghir [34] for CPVC pipe fitting material.

The linear dependence of elastic modulus on temperature at different crosshead speeds can be expressed as:

$$E = 3238.3 - 14.81\, T\,(^{o}\mathrm{C}) \qquad \text{for } v = 5 \text{ mm/min} \tag{4.16}$$

$$E = 3440.7 - 17.88\, T\,(^{o}\mathrm{C}) \qquad \text{for } v = 50 \text{ mm/min} \tag{4.17}$$

$$E = 3602.1 - 17.44\, T\,(^{o}\mathrm{C}) \qquad \text{for } v = 500 \text{ mm/min} \tag{4.18}$$

By comparing the slopes, it can be seen that the effect of temperature on elastic modulus at different crosshead speeds are almost similar.

Figure 4.13 shows the variation of elastic modulus with temperature for CPVC at room temperature and a crosshead speed of 5 mm/min. The values of E from Merah et al. [35] for CPVC pipe fittings at 5 mm/min, Povolo et al. [27] for PVC at $6.6\text{x}10^{-4}$ s^{-1} and

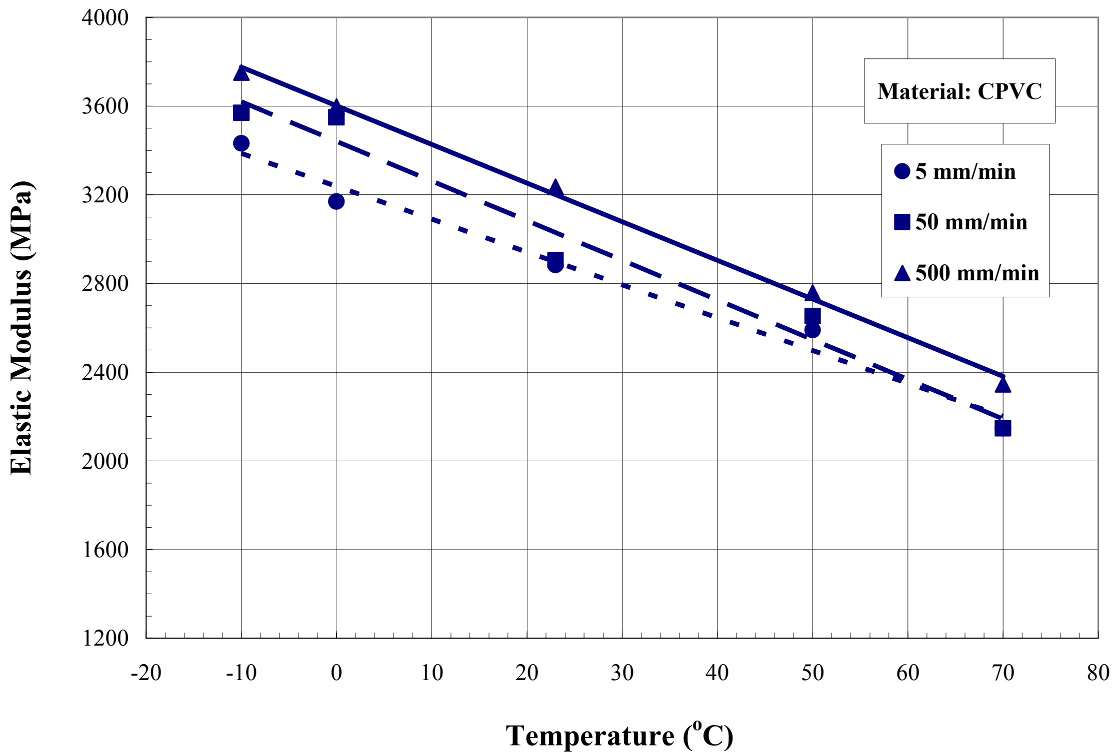


Figure 4.12 Effect of temperature on elastic modulus at three different crosshead speeds.

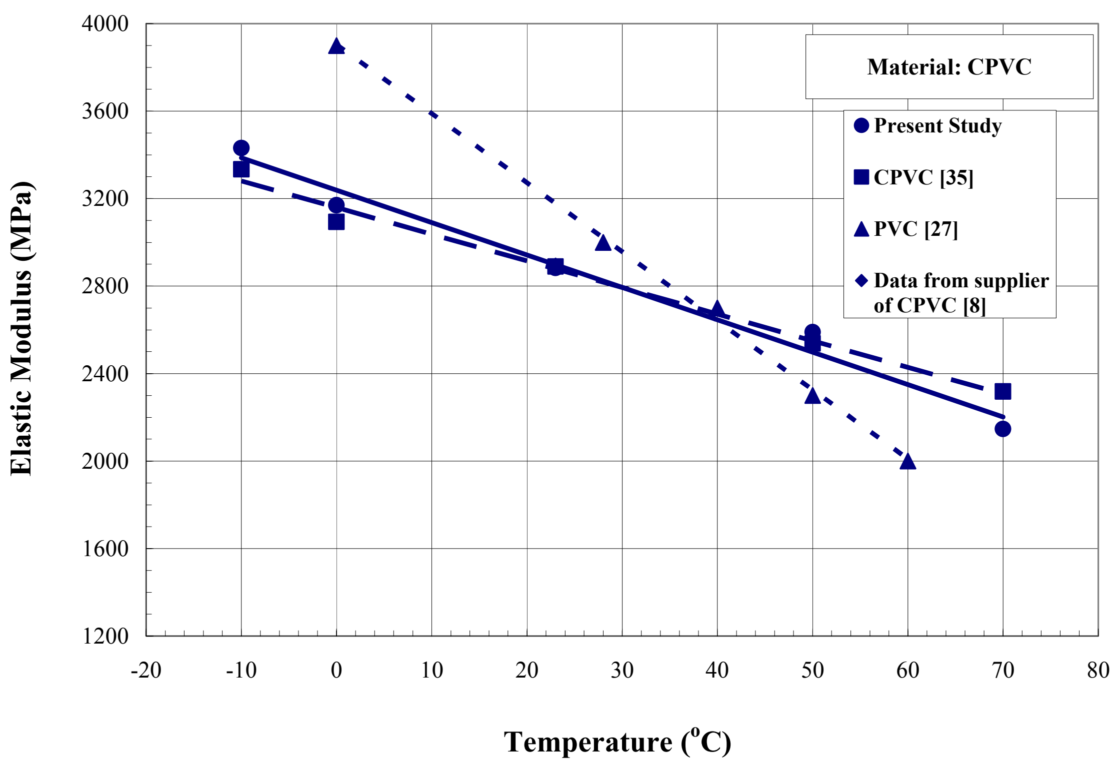


Figure 4.13 Variation of elastic modulus with temperature at low crosshead speed.

supplier of CPVC [8] are also shown in the figure. The comparison between the temperature dependence of PVC and CPVC reveals that in high temperature range, the elastic modulus for PVC decreases at a faster rate than that of CPVC. Povolo et al. [27] found that the data of modulus of elasticity had a linear dependence with temperature for PVC, which was expressed as:

$$E(T) = E_o - \xi T \tag{4.19}$$

with E_o = 13.3 GPa and ξ = 34 MPa/K at a strain rate of 6.6x10^{-4} s^{-1}. Merah et al. [35] found that the values of E_o and ξ for CPVC coupling material at 5 mm/min were 6.53 GPa and 12.40 MPa/K, respectively.

4.4 Fracture Strain

Fracture strain is defined as the strain at which specimens break. It is a measure of ductility and it is obtained from the length of fracture, L_f, of the gage section that originally had length L_i:

$$\varepsilon_f = (L_f - L_i)/ L_i \tag{4.20}$$

The fracture strain corresponds to the same point on the stress-strain curve as the fracture strength σ_f [13].

The qualitative description of the variation of fracture strain with crosshead speed and temperature is shown in figure 4.14. The fracture strain remains almost constant at room temperature and below. At 50 and 70^{o}C, an enhancement of ε_f occurs with increasing crosshead speed between 5 and 50 mm/min then stabilized with further increasing crosshead speed. For the effect of temperature on fracture strain, ε_f, first increases slowly with increasing temperature in the temperature range of -10 to 50^{o}C at all crosshead speeds. However, ε_f increases rapidly between 50 and 70^{o}C at moderate and high crosshead speeds, going from 10.76 to 37.39% at 50 mm/min and from 17.77 to 35.44% at 500 mm/min.

4.4.1 Effect of Strain Rate on Fracture Strain

Figure 4.15 shows the variation of fracture strain for CPVC with crosshead speed at different temperatures. It can be seen that the value of fracture strain remains almost constant at low temperatures (-10, 0 and 23^{o}C) with increasing crosshead speed. This is mainly because the molecular chains were frozen from moving at low temperatures. However, ε_f increases at the temperature of 50 and 70^{o}C when the crosshead speed is increased from 5 to 50 mm/min then stabilizes with further increasing strain rate. At 70^{o}C, ε_f increases rapidly because of enhancement of molecular mobility. Ductile fracture is expected at this temperature.

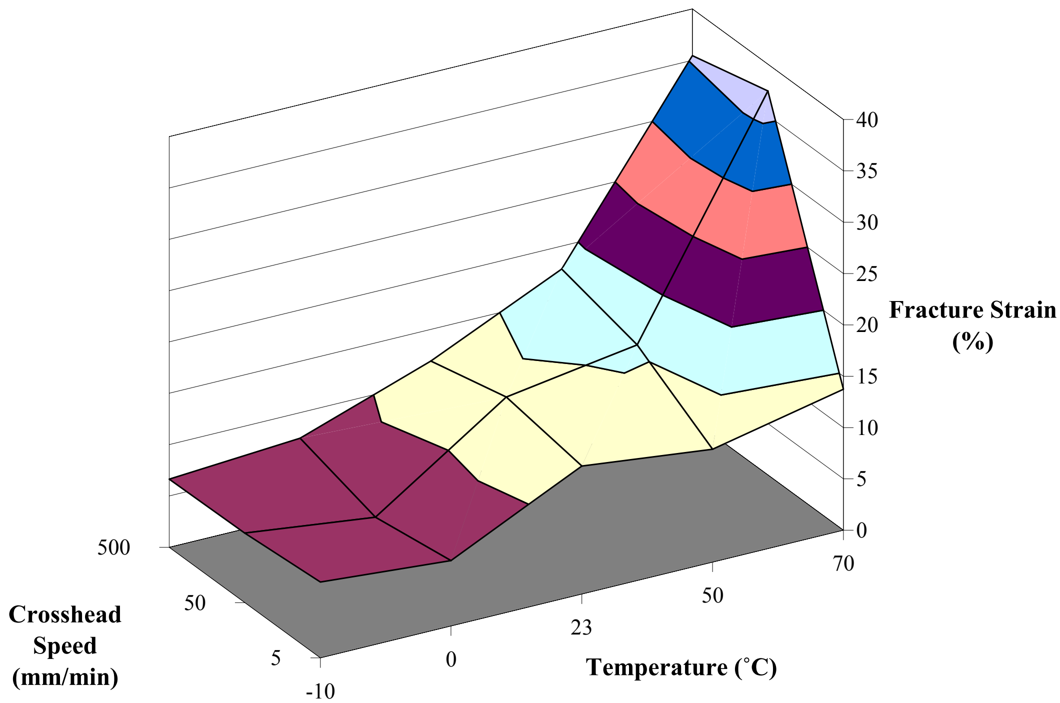


Figure 4.14 Variation of fracture strain with crosshead speed and temperature (not to scale).

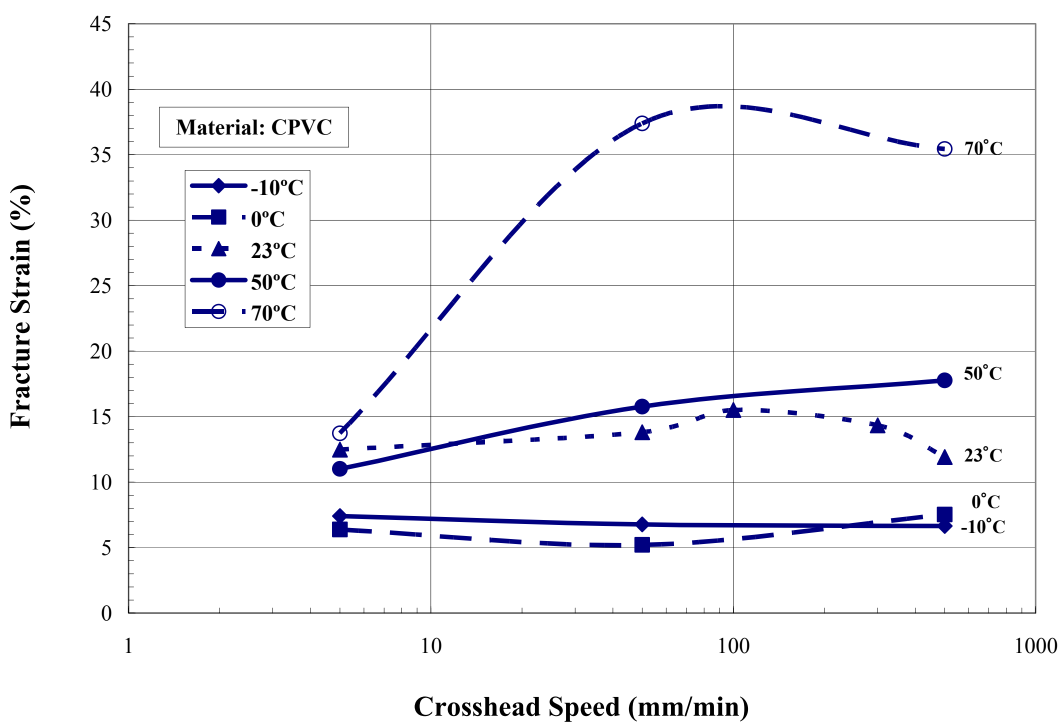


Figure 4.15 Fracture strain dependence of crosshead speed at different temperatures.

4.4.2 Effect of Temperature on Fracture Strain

Fracture strain or ductility generally increases with temperature. Merah et al. [35] found that the fracture strain for CPVC pipe fitting material increased with increasing temperature in the temperature range (-10 to 70^{o}C) at 5 mm/min. Similar behavior was found by Andrews [1] for PMMA. Figure 4.16 shows the variation of fracture strain with temperature at different crosshead speeds. The fracture strain generally increases with temperature at low crosshead speed and the effect is minor. At 50 and 500 mm/min, the curves display two distinct regions. In the first one (-10 to 50^{o}C), the fracture strain increases slowly with increasing temperature. In the second region, however, ε_f-T slope increases rapidly between 50 and 70^{o}C. This enhancement of fracture strain occurred with increasing molecular mobility that allows easier chain disentanglement resulting in their alignment and leading to higher plastic deformation. Ductile fracture is expected in this region. The results obtained at low crosshead speed are compared to those obtained by Merah et al. [35] for CPVC and Andrews [1] for PMMA over the temperature range of -10 to 70^{o}C as shown in figure 4.17. It can be seen that the temperature has a similar effect on these materials.

At 70^{o}C, considerable necking is observed to occur at moderate and high crosshead speeds where the fracture strains are 38.80 and 36.21%, respectively. The neck starts to form at the point where the cross-sectional area of the specimen is minimum. Once the whole cross-section has yielded, the neck travels along the length of the specimen at constant load until fracture occurs.

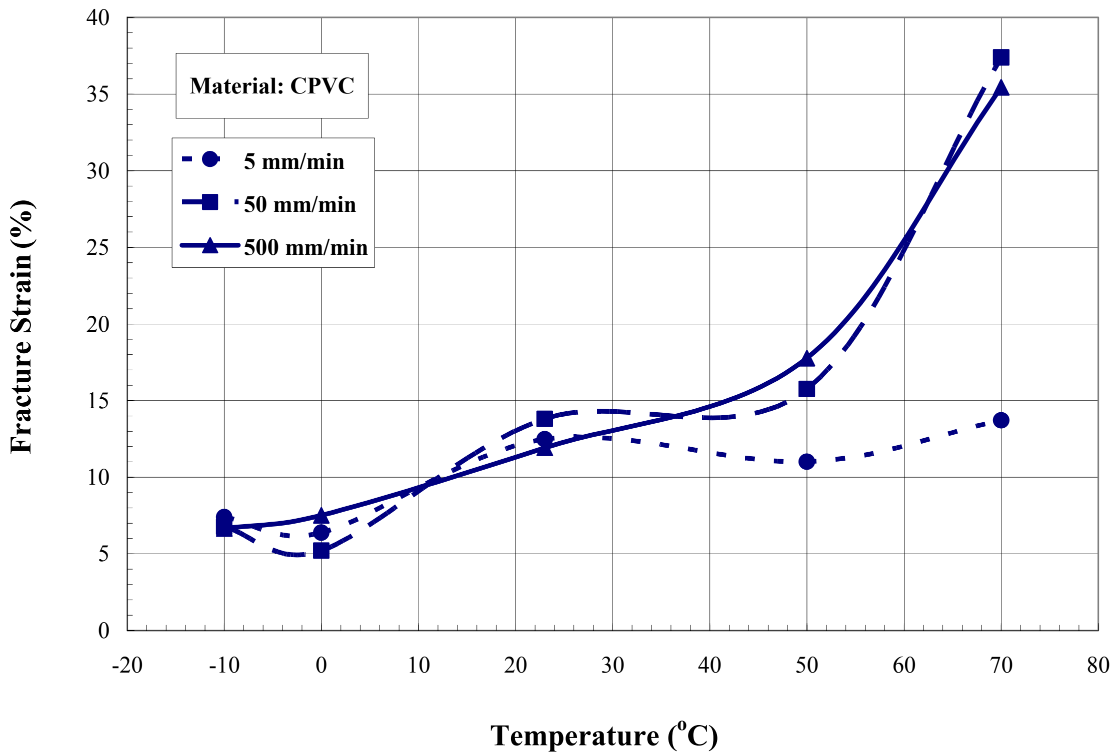


Figure 4.16 Variation of fracture strain with temperature at different crosshead speeds.

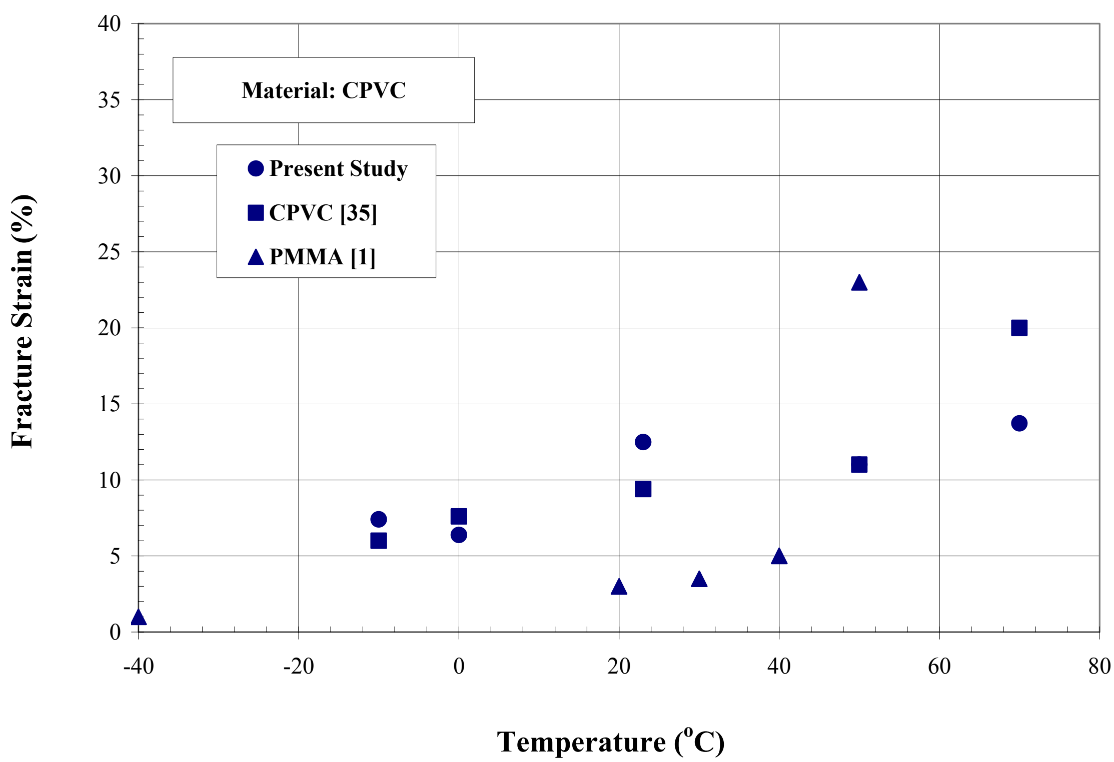


Figure 4.17 Variation of fracture strain with temperature at low crosshead speed.

CHAPTER 5

EFFECTS OF STRAIN RATE AND TEMPERATURE ON FRACTURE TOUGHNESS

This chapter begins with the theoretical background about fracture toughness. Next, the measurement and plane strain validity of fracture toughness are discussed. After that, the effects of strain rate (crosshead speed) and temperature on fracture toughness will be presented. The effects of strain rate and temperature on crack tip blunting and crack tip plastic zone will be then discussed. An attempt will be made to correlate fracture toughness and fracture modulus. Finally, the analysis of fracture surfaces of tested specimens at different crosshead speeds and temperatures will be presented and used to explain the different effects.

5.1 Theoretical Background

The application of fracture mechanics parameter, fracture toughness, to describe and characterize crack growth in polymers was established in 1970 [36]. Fracture mechanics is the science that relates the strength of a fractured structure to the material's fracture toughness and to the crack size, shape, location and sharpness. Test methods that measure a material's fracture toughness are necessary for safe design and for accurate failure analysis. Engineers and researchers who tried to measure fracture toughness of plastics have relied almost exclusively on metals testing technology. In some cases, metals fracture testing technology is inadequate on theoretical grounds [11].

When designing a structure against fracture, there are three critical variables that must be considered: stress, flaw size and fracture toughness. Figure 5.1 illustrates the fracture mechanics triangle. Fracture mechanics provides a mathematical relationship between these quantities. In most cases there are two degrees of freedom; a knowledge of two quantities is required to compute the third [11].

For thin specimens, the value of fracture toughness K_c depends on and decreases with increasing specimen thickness, as indicated in figure 5.2. However, K_c becomes independent of thickness when the behavior is plane strain. For thicker specimens, K_c is known as the plane strain fracture toughness K_{IC} which is defined as:

$$K_{IC} = Y\sigma\sqrt{\pi a} \tag{5.1}$$

The plane strain fracture toughness K_{IC} is a fundamental material property that depends on many factors, the most influential of which are temperature, strain rate and microstructure. The magnitude of K_{IC} diminishes with increasing strain rate and decreasing temperature. Furthermore, an enhancement in yield strength brought by solid solution or dispersion additions or by strain hardening generally produces a corresponding decrease in K_{IC}. Furthermore, K_{IC} normally increases with reduction in grain size as composition and other microstructural variables are maintained constant [9].

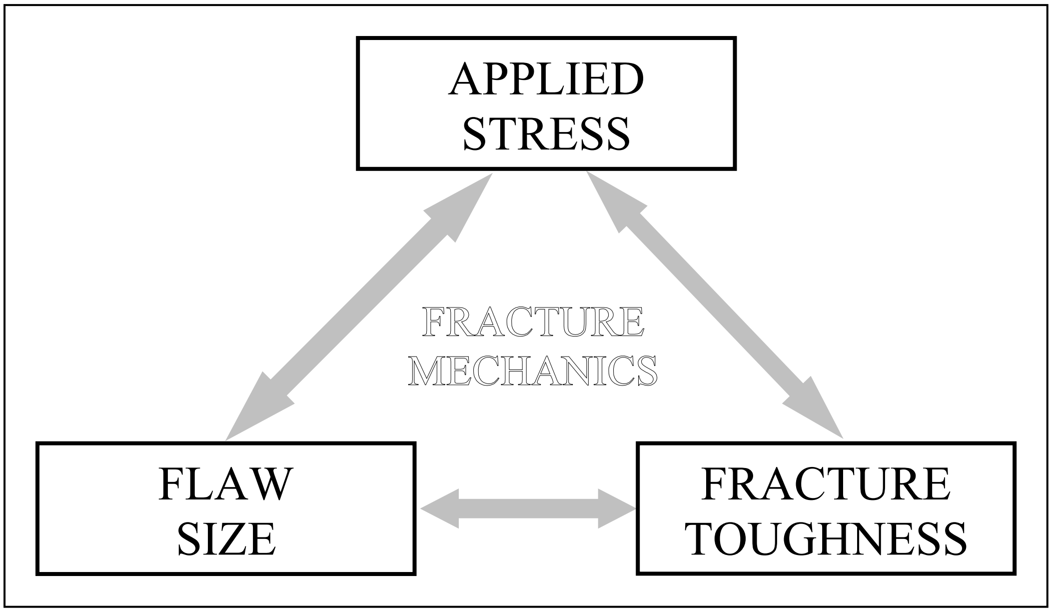


Figure 5.1 The fracture mechanics triangle, which identifies the three critical variables in fracture design [11].

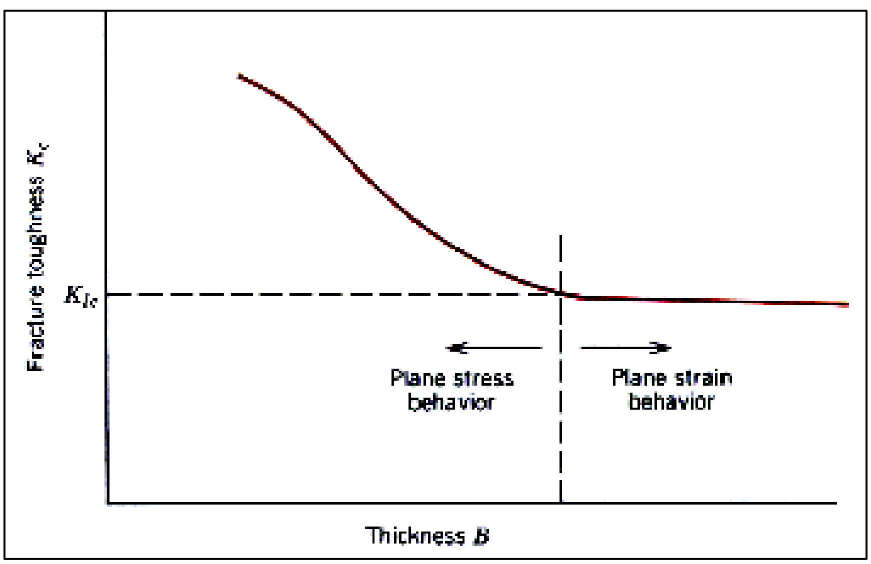


Figure 5.2 Schematic representation showing the effect of plate thickness on fracture toughness [9].

5.2 Fracture Toughness Measurement

The single-edge-notch bending specimens which were prepared, as outlined in section 3.1, were tested on Instron 8801 machine at five temperatures (-10, 0, 23, 50 and 70^{o}C) and three crosshead speeds (5, 50 and 500 mm/min). In fracture toughness testing, an increasing displacement is applied to pre-cracked specimens of CPVC until they fracture. During testing, the data of applied load *P* and crack opening displacement *COD*, measured using Instron COD gauge, were recorded using the Instron BlueHill Series machine control data acquisition and analysis software. Representation of load-displacement curves for different crosshead speeds at -10, 0, 23, 50 and 70^{o}C are shown in figures 5.3 through 5.7, respectively. It is noticed that in general, the applied maximum load increases with decreasing crosshead speeds at low temperatures. However, it increases with increasing crosshead speeds above room temperature. Also, it is observed that the applied maximum load increases with increasing temperature up to the temperature of 50^{o}C then it decreases at 70^{o}C.

The critical load, P_Q, was defined in one of several ways, depending on the type of load-displacement curves as shown in figure 5.8. The 5% secant line (i.e. a line from the origin with a slope equal to 95% of the initial slope) is constructed to determine P_5. In the case of Type I behavior, the load-displacement curve is smooth and it deviates slightly from linearity before ultimate failure at P_{max}. This nonlinearity can be caused by plasticity, subcritical crack growth, or both. For a Type I curve, P_Q is equal to P_5. With a Type II curve, a small amount of unstable crack growth (i.e. a pop-in) occurs before the curve deviates from linearity by 5%. In this case P_Q is defined at the pop-in. A specimen

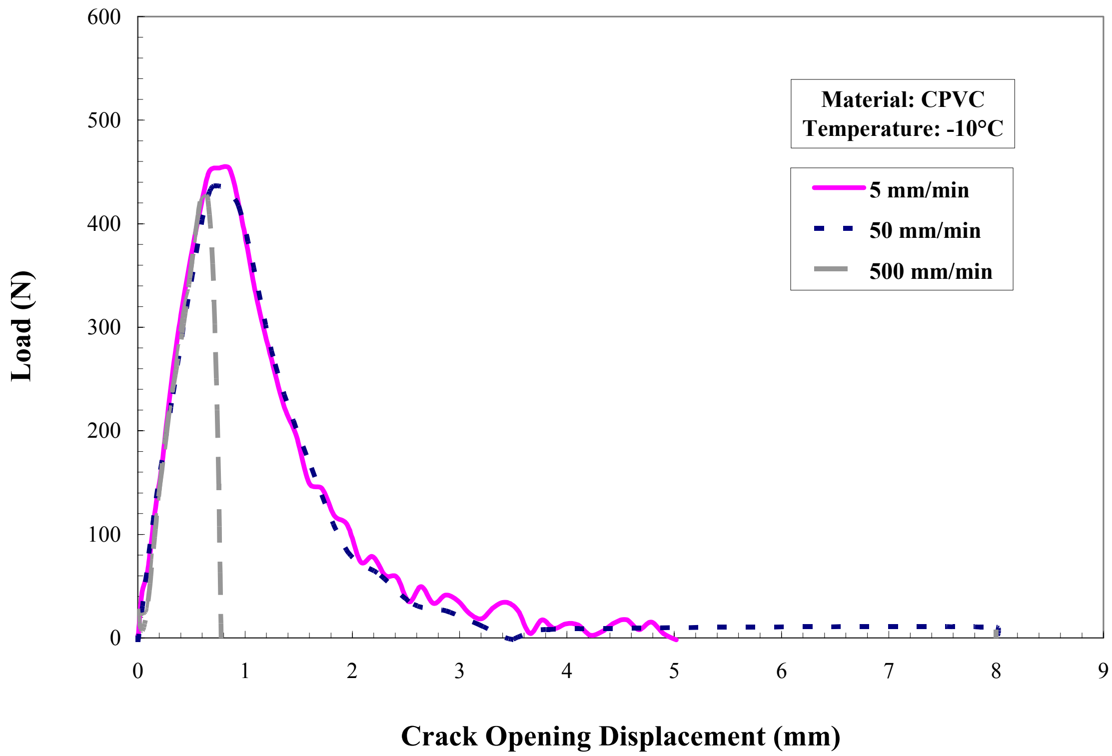


Figure 5.3 Load-*COD* curves at -10^{o}C for different crosshead speeds.

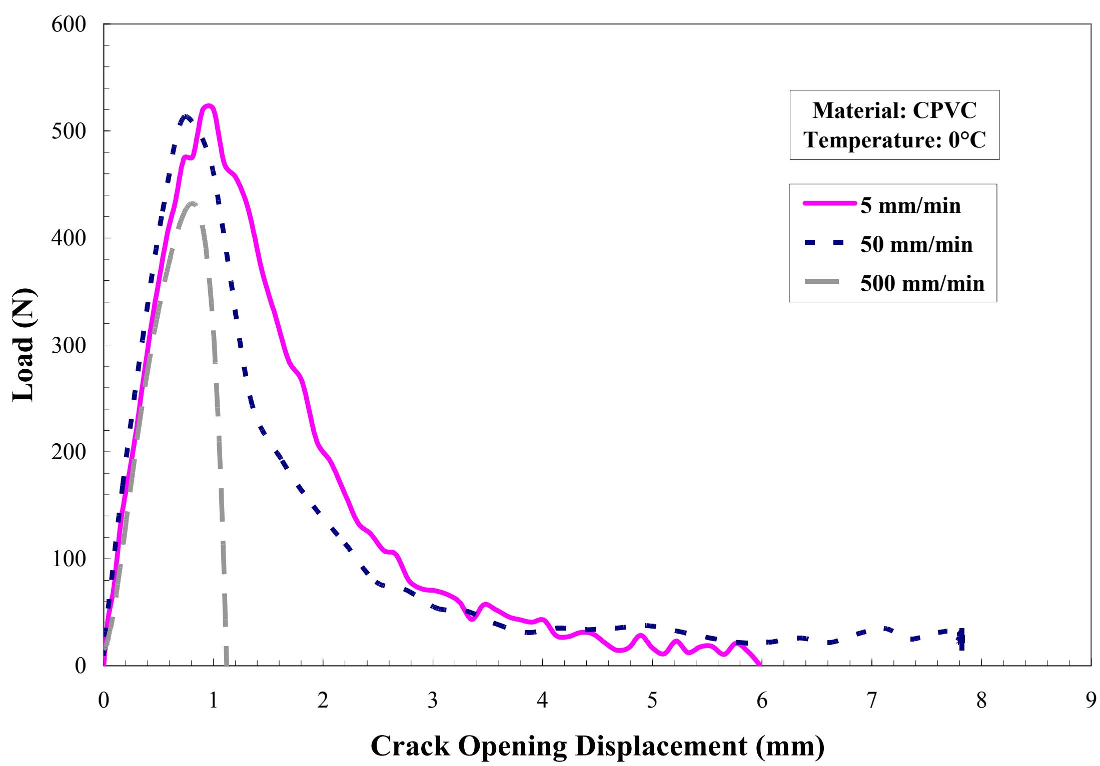


Figure 5.4 Load- *COD* curves at 0^{o}C for different crosshead speeds.

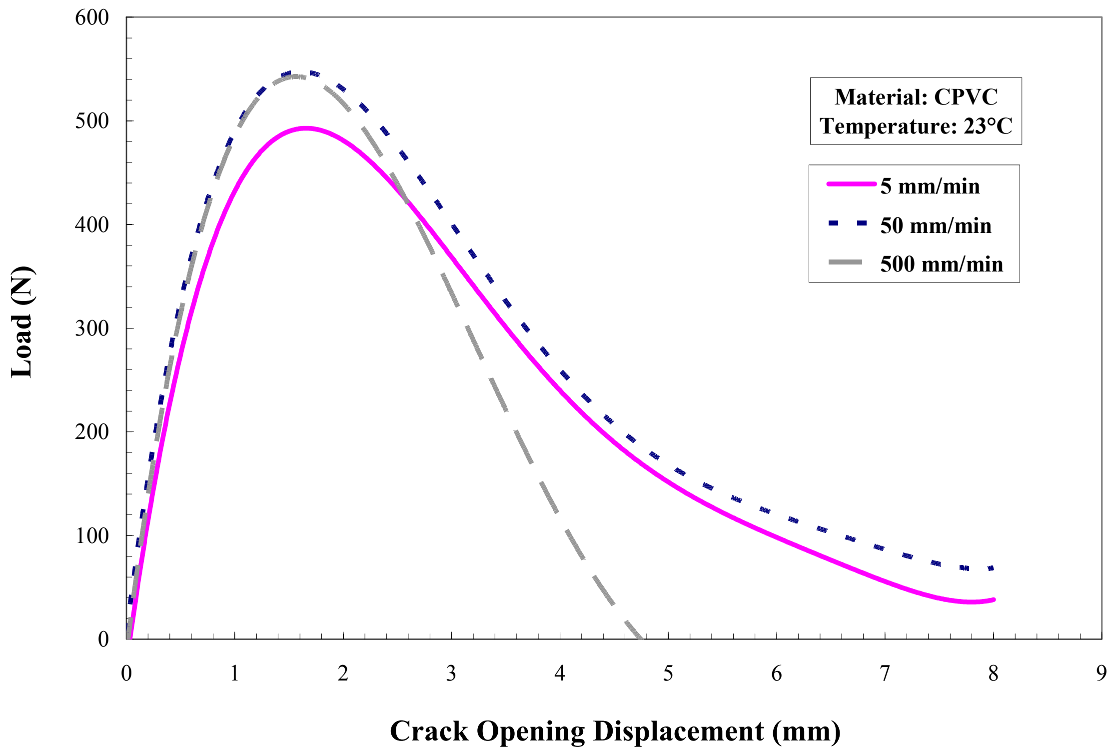


Figure 5.5 Load- *COD* curves at 23^{o}C for different crosshead speeds.

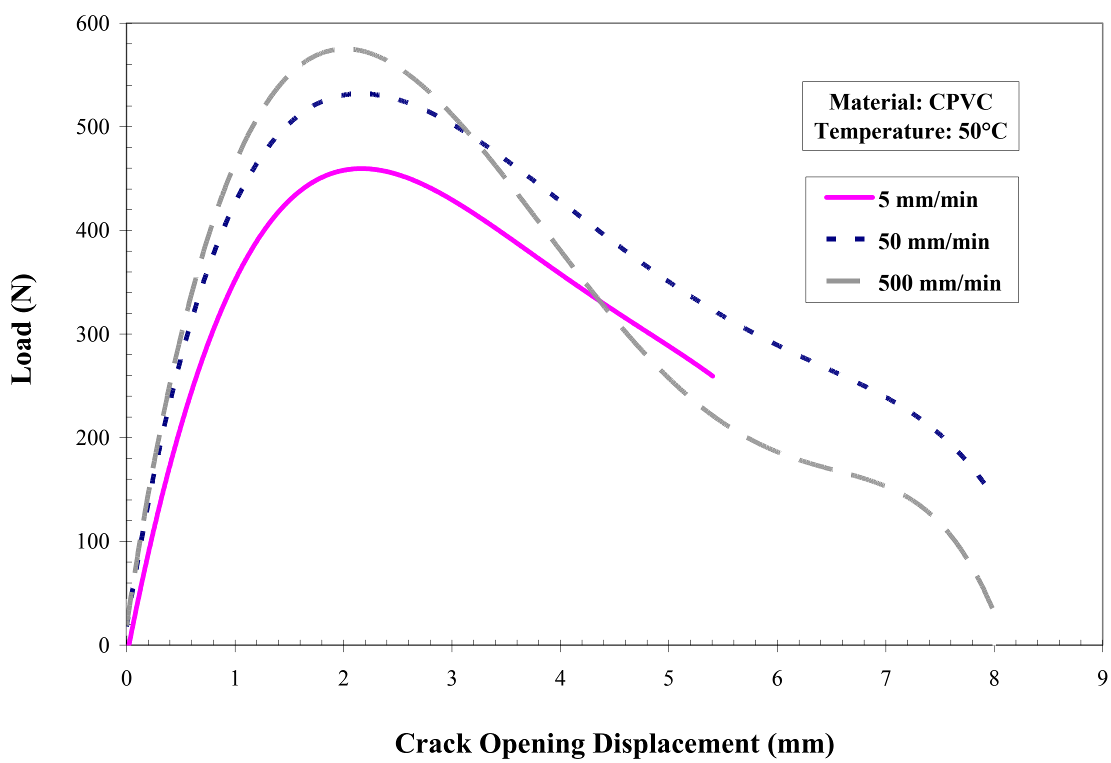


Figure 5.6 Load- *COD* curves at 50^{o}C for different crosshead speeds.

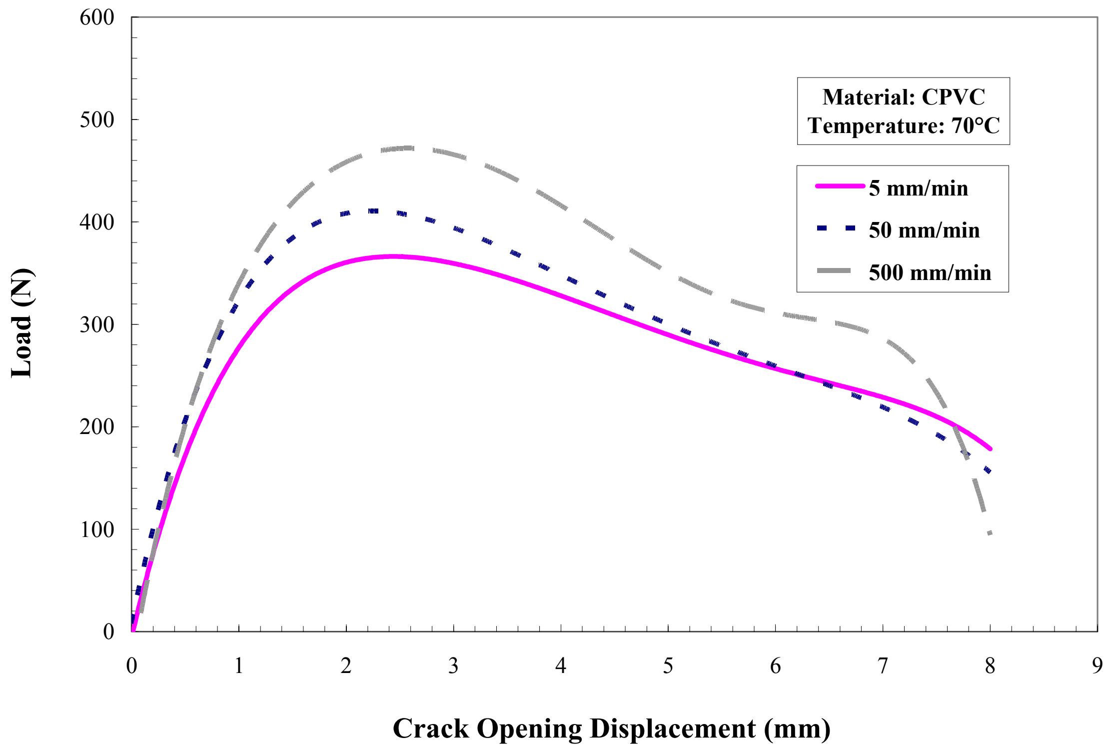


Figure 5.7 Load- *COD* curves at 70°C for different crosshead speeds.

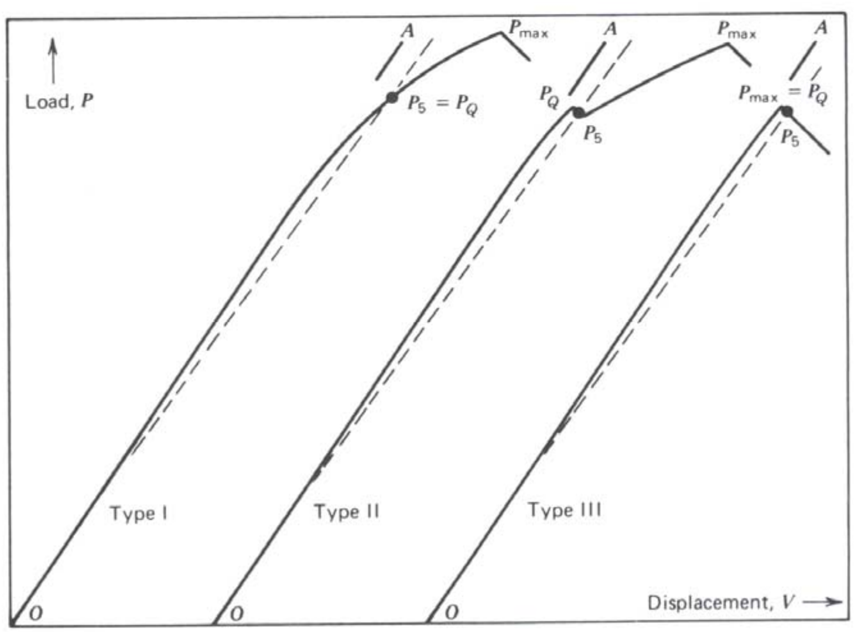


Figure 5.8 Schematic representation showing the effect of plate thickness on fracture toughness [48].

that exhibits Type III behavior fails completely before achieving 5% nonlinearity. In such case, $P_Q = P_{max}$ [48].

Figure 5.9 shows the critical load of Load-*COD* curves at 0°C for different crosshead speeds. The average values of the critical load for the three tests performed at each condition for different crosshead speeds and temperatures are illustrated in Table 5.1. At 5 mm/min, the critical load increases with increasing temperature up to 23°C then reduces with further increasing temperature. For 50 and 500 mm/min, however, P_Q increases with temperature up to 50°C then decreases at 70°C. Consequently, the fracture toughness will follow a similar behavior according to the relationship between P_Q and K_Q.

Once P_Q is determined, the fracture toughness, K_Q, is computed from the following relationship according to ASTM D5045-93 [46]:

$$K_Q = \frac{P_Q S \cdot f(a/W)}{BW^{3/2}} \tag{5.2}$$

where *f(a/W)* is a dimensionless function of *a/W* and calculated by:

$$f(a/W) = \frac{3(a/W)^{1/2}[1.99 - (a/W)(1 - a/W)(2.15 - 3.93(a/W) + 2.7(a/W)^2)]}{[2(1 + 2(a/W))(1 - (a/W))^{3/2}]} \tag{5.3}$$

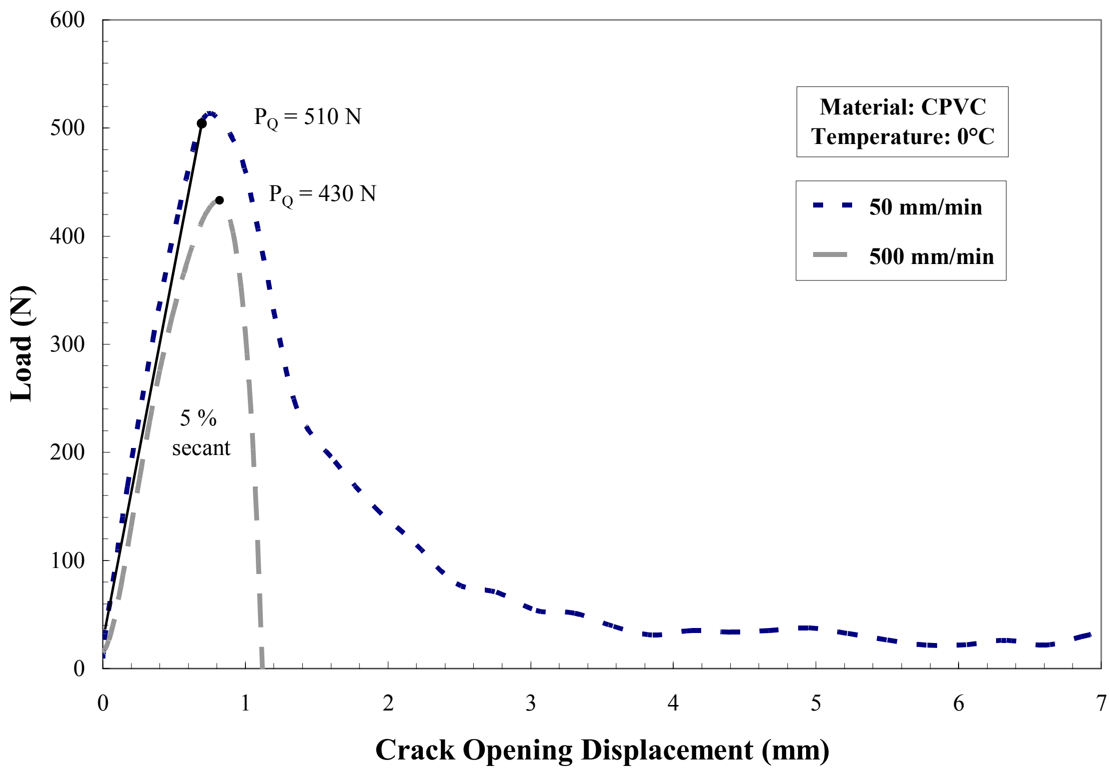


Figure 5.9 The critical load of Load-*COD* curves at 0^{o}C for different crosshead speeds.

Table 5.1 Average values of the critical load P_Q (N) as a function of crosshead speed and temperature.

Temp. / **v (mm/min)**	**-10ºC**	**0ºC**	**23ºC**	**50ºC**	**70ºC**
5	440	463	487	452	357
50	452	505	512	517	422
500	440	447	503	568	453

and

S = loading span, $S = 4W = 76$ mm (see figure 5.10)

a = specimen crack length = 9 mm

W = specimen width = 19 mm

B = specimen thickness = 9.5 mm (identical to the pipe thickness)

The fracture toughness values, K_Q, were computed using equations 5.2 and 5.3. The average values for the three tests performed at different crosshead speeds and temperatures are illustrated in Table 5.2.

The tentative values of fracture toughness, K_Q, based on a graphical construction of the load-crack opening displacement curve, are considered a valid K_{IC}, only if they satisfy the following conditions:

$$0.45 < a / W < 0.55 \tag{5.4}$$

$$\frac{P_{\max}}{P_Q} < 1.1 \tag{5.5}$$

and

$$B, a, W - a > C \tag{5.6}$$

where $C = 2.5 * \left(\frac{K_Q}{\sigma_y}\right)^2$ and σ_y is the yield stress for the temperature and loading rate of each test. These criteria require that thickness B must be sufficient to ensure plane strain

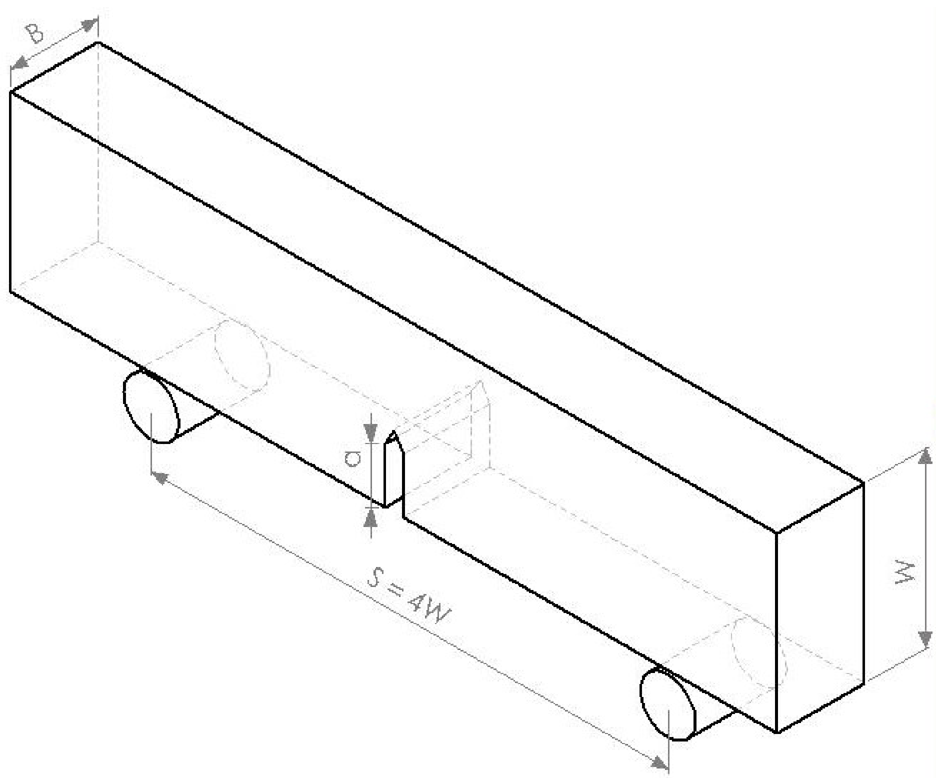


Figure 5.10 Schematic illustration of a single-edge-notch three-point bend specimen.

Table 5.2 Average values of the fracture toughness K_Q (MPa.m$^{1/2}$) as a function of crosshead speed and temperature.

Temp. / v (mm/min)	-10ºC	0ºC	23ºC	50ºC	70ºC
5	3.43	3.74	3.94	3.65	2.98
50	3.69	3.87	4.18	4.24	3.40
500	3.55	3.62	3.95	4.69	3.69

and that ligament width ($W - a$) be sufficient to avoid excessive plasticity in the ligament. Equation 5.5 restricts the amount of error possible between the actual value of load at the moment of crack extension and that obtained graphically from the load-displacement record.

According to equations 5.4 and 5.5, the first two requirements for the validity of K_{IC} for each individual condition are met in the present study. However, the third criterion based on equation 5.6 is not always satisfied. Tables 5.3 through 5.7 summarize the validity of this criterion for the temperatures of -10, 0, 23, 50 and 70^{o}C, respectively where "Y" indicates the criterion is met and "N" indicates the criterion is not met. For -10^{o}C, the size criterion was met for all dimensions at different crosshead speeds and the plane strain fracture toughness is valid. For 0^{o}C, the criterion was met for some dimensions in most cases and $K_Q = K_{IC}$ at the crosshead speed of 500 mm/min. However, the criterion was not met for all dimensions in all cases for the temperatures of 23, 50 and 70^{o}C and $K_Q \neq K_{IC}$.

The validity of plane strain fracture toughness was discussed by Ye et al. [33] for TPX polymer at crosshead speed of 0.5, 5, 50 and 500 mm/min at ambient temperature. The plane strain condition was not satisfied for some dimensions of SENB specimen in most cases. Cayard [49] determined the appropriate specimen size requirements necessary for a valid prediction of fracture toughness on the rigid PVC using linear elastic fracture mechanics (LEFM) approach. He found that the requirements were never satisfied in the fracture toughness tests conducted at room temperature.

Anderson [11] stated that because the size requirements of ASTM E399-90 [50], K_{IC} standard for metals which is very similar to ASTM D5045-93 [46], are very stringent,

Table 5.3 Validity of specimen dimensions for Plane Strain Fracture Toughness at -10^{o}C.

Requirement / **v (mm/min)**	***B > C***	***a > C***	***W - a > C***
5	Y	Y	Y
50	Y	Y	Y
500	Y	Y	Y

Table 5.4 Validity of specimen dimensions for Plane Strain Fracture Toughness at $0^{o}C$.

Requirement / v (mm/min)	$B > C$	$a > C$	$W - a > C$
5	N	N	N
50	Y	N	Y
500	Y	Y	Y

Table 5.5 Validity of specimen dimensions for Plane Strain Fracture Toughness at 23^{o}C.

Requirement / v (mm/min)	***B* > *C***	***a* > *C***	***W* - *a* > *C***
5	N	N	N
50	N	N	N
500	N	N	N

Table 5.6 Validity of specimen dimensions for Plane Strain Fracture Toughness at $50^{o}C$.

Requirement / **v (mm/min)**	***B > C***	***a > C***	***W - a > C***
5	N	N	N
50	N	N	N
500	N	N	N

Table 5.7 Validity of specimen dimensions for Plane Strain Fracture Toughness at 70^{o}C.

Requirement / **v (mm/min)**	***B* > *C***	***a* > *C***	***W* - *a* > *C***
5	N	N	N
50	N	N	N
500	N	N	N

it is difficult and sometimes impossible to measure a valid K_{IC} in most structural materials. A material must either be relatively brittle or the test specimen must be very large for linear elastic fracture mechanics to be valid. In the present case, the specimen size is constrained by the thickness of the pipe (9.5 mm) from which it was produced. Thus, the size requirement is not met for temperatures where ductile fracture is expected. In addition, this is a comparative study and that the validity of all criteria need not be satisfied. However, this proves that when material behaves in ductile manner fracture toughness specimen size is important.

5.3 Effect of Strain Rate on Fracture Toughness

Strain rate (crosshead speed) has a significant effect on the fracture toughness of many polymers. This effect has been investigated by Han et al. [14] on PEI, Marshal and co-workers [38, 41] on PMMA and PS, Ye et al. [33] on TPX, Karger-Kocsis and Friedrick [39] on PEEK and Merah et al. [45] on CPVC pipe fitting material at different testing conditions.

The effect of crosshead speed on the value of fracture toughness measured in the present study (Table 5.2) at different temperatures is illustrated in figure 5.11. These results reveal that the average value of fracture toughness for the temperature of -10, 0 and 23^{o}C increases slightly when the crosshead speed is increased from 5 to 50 mm/min; for example from 3.94 to 4.18 MPa.m$^{1/2}$ for 23^{o}C. For the range of 50 to 500 mm/min, however, it decreases slightly with increasing crosshead speed. The reduction of K_Q at

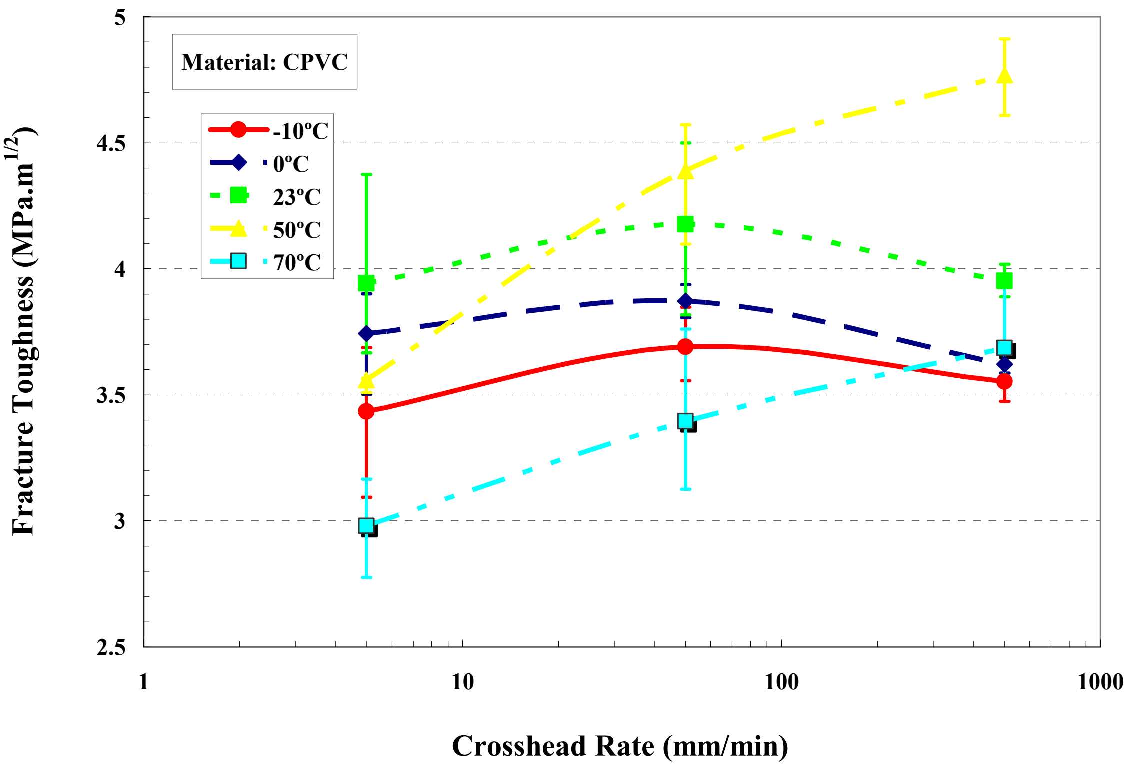


Figure 5.11 Effects of crosshead speed on fracture toughness of CPVC.

high crosshead speed can be explained by a reduced molecular mobility or a reduced plastic flow zone at high crosshead speeds and thus a low ductility is expected. This result is consistent with the observed increase in the yielding stress when the crosshead speed is increased up to 500 mm/min. The maximum value of K_Q at the temperatures of -10, 0 and 23^{o}C is 3.69, 3.87 and 4.18 MPa.m$^{1/2}$ respectively, observed near v = 50 mm/min. The reason why CPVC polymer achieves the highest fracture toughness at a moderate crosshead speed rather than at lower speed is not clear. But similar trend of the fracture toughness as a function of crosshead speed was also observed by Ye et al. [33] for TPX polymer using SENB specimens, Karger-Kocsis and Friedrick [39] for PEEK using CT specimens and Han et al. [14] for PEK-C using SENB specimens.

The results obtained at 50 and 70^{o}C show that the value of K_Q increases with crosshead speeds. This is because of enhancement of fracture strain, crack tip blunting and plastic zone size at high temperatures (50 and 70^{o}C). However, the results obtained at 70^{o}C show K_Q values are the lowest at crosshead speeds of 5 and 50 mm/min. Similar results were found by Marshal et al. [39, 41] for PMMA and PS at 20^{o}C using SEN fatigue specimens. Further investigation of the effect of crosshead speed on fracture toughness will be discussed in section 5.5, 5.6 and 5.8.

5.4 Effect of Temperature on Fracture Toughness

The environmental temperature has a considerable effect on fracture toughness of thermoplastics because these polymers exhibit a transition from brittle to ductile as testing temperature increases. Figure 5.12 illustrates the influence of temperature on the fracture toughness, K_Q, at three different crosshead speeds (v = 5, 50 and 500 mm/min). In general, the three curves show a maximum K_Q value at temperatures which seem to increase with increasing crosshead speed. At low crosshead speed (v = 5 mm/min), it can be seen that the value of fracture toughness increases with temperature and reaches a maximum value of 3.94 at room temperature then decreases to a minimum value of 2.98 MPa.m$^{1/2}$ at 70^{o}C. Similar general behavior is observed at moderate and high crosshead speeds (v = 50 and 500 mm/min). The maximum value of fracture toughness at 50 mm/min is around 40^{o}C while that at 500 mm/min is about 50^{o}C. This change in fracture toughness is normally associated with a transition from brittle to ductile fracture. These results suggest that the transition from brittle to ductile fracture occurs at higher temperature for high loading rate. The transition temperature increases with crosshead speed because of the enhancement in brittleness of CPVC as crosshead speed increases. It should be mentioned that all these transition temperatures are way below the glass transition temperature (T_g) of the CPVC material used in this investigation. As shown in figure 5.13, the Differential Scanning Calorimetry (DSC) measured T_g was about 120^{o}C.

Similar results were observed by several investigators. Han et al. [14] found that the PEK-C material increased with temperature and peaked at 160^{o}C then dropped off with further increasing temperature. T_g for PEK-C was 215^{o}C. Ye et al. [33] found that the fracture toughness value for TPX reached a maximum value at 60^{o}C at high crosshead

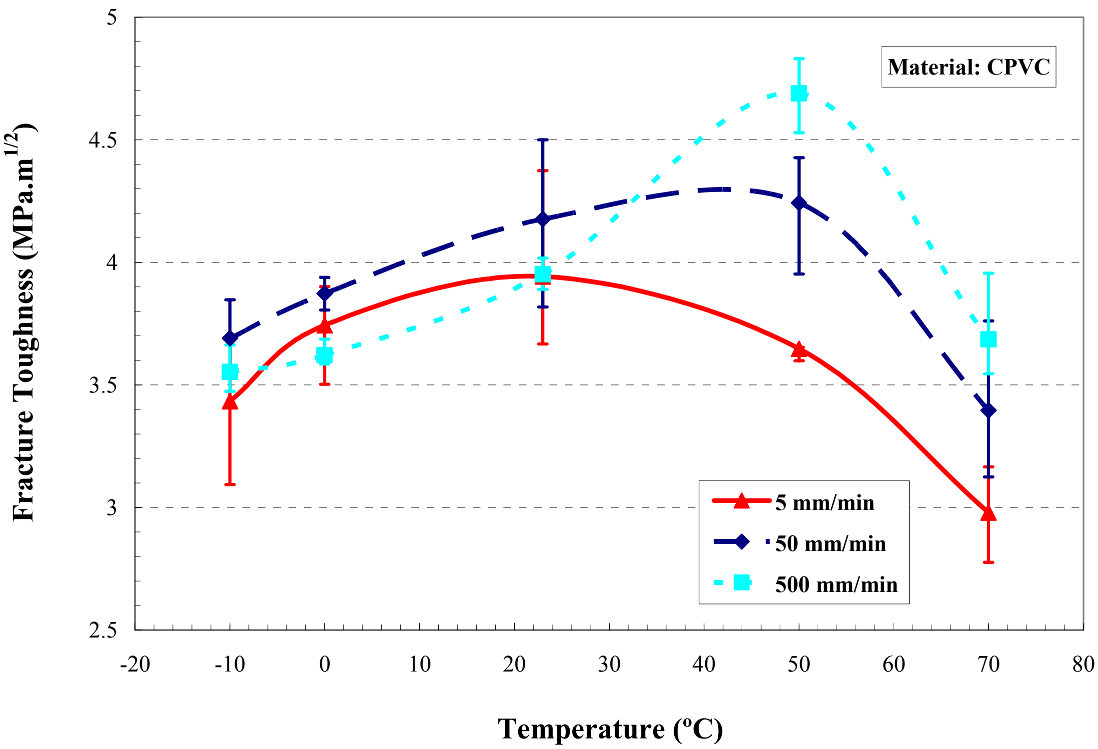


Figure 5.12 Effects of temperature on fracture toughness at different crosshead speeds.

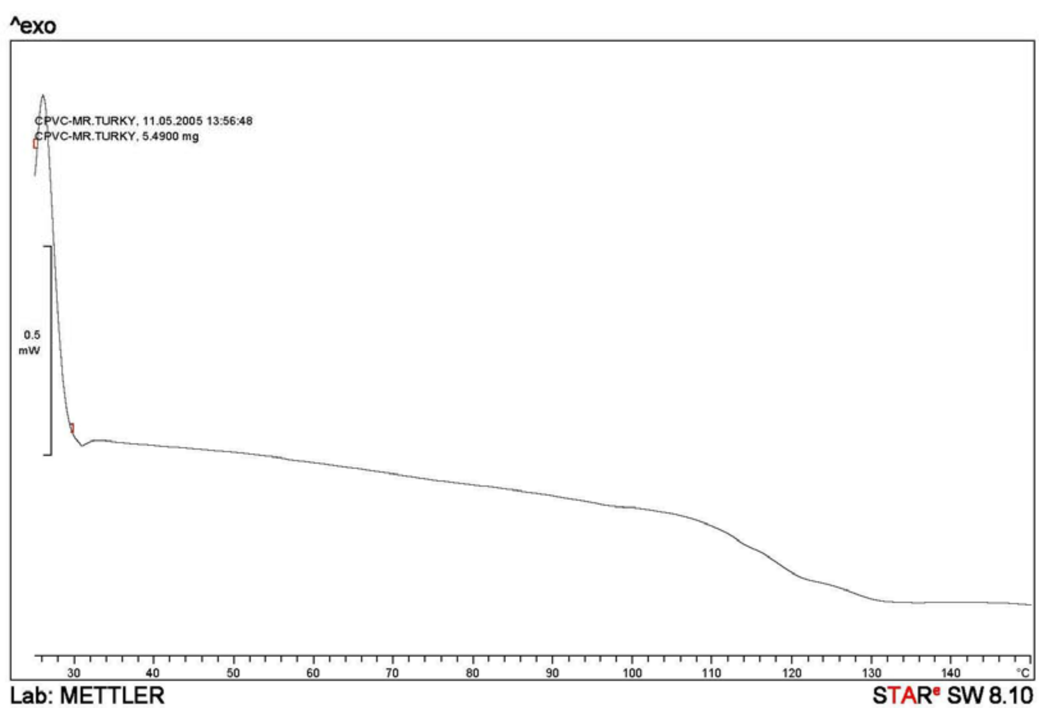


Figure 5.13 Glass transition temperature of CPVC.

rate of 500 mm/min then decreased with increasing temperature. This temperature was associated with a transition from brittle fracture to ductile-viscous fracture whereas the T_g for TPX was 81°C. Karger-Kocsis and Friedrich [39] concluded that the change in fracture toughness for PEEK at 115°C was associated with a plane strain-plane stress transition causing a change from brittle to ductile failure. The glass transition temperature for PEEK was 144°C. Others [51] stated that the transition temperature shifted down may be attributed to local adiabatic heating at the crack tip, due to the very rapidly introduced mechanical energy around the crack tip.

From the literature, it is difficult to infer the obvious generality of the temperature dependence of polymer fracture toughness because fracture behavior is highly complicated due to their interrelated dependence on testing variables and material properties. Kim and Ye [23] stated that "the fracture behavior changes from brittle to ductile, or vice versa in response to not only temperature, but also other numerous variables such as notch tip radius, specimen thickness and geometry, loading mode and rate, molecular weight, crystallinity, physical aging, etc.".

In an attempt to shed some light on this subject crack tip blunting and plastic zone size are measured and the results are analyzed in the following sections.

5.5 Crack Tip Blunting

Crack tip blunting is defined in terms of crack tip opening displacement (*CTOD*) which is a measure of fracture toughness. The concept of crack tip blunting was originally proposed by Kinloch and Williams [52] to explain unstable "stick-slip" fractures observed in epoxies under quasi-static conditions. Blunting occurs as a direct result of plastic shear flow at the crack tip. Han et al. [14] observed a "stretched zone" formed at the crack initiation in PEK-C tested with SENB specimens at ambient temperature. The "stretched zone" can only be formed if there is plastic flow and blunting of the sharp crack tip.

In this study, it was observed that at -10 and 0^{o}C, the temperature rise is not sufficient to cause crack tip blunting. Figure 5.14 shows a photograph of SENB specimen at crosshead speed of 50 mm/min at the temperature of -10^{o}C. It can be seen that the crack is sharp and the plastic zone size is very small even for this long crack. However, it is observed that blunting occurs at the crack tip of specimens tested at room temperature and above. The *CTOD* can be estimated by assuming that the SENB specimen halves are rigid and rotate about a hinge point:

$$\delta = \frac{r(W-a)V}{r(W-a)+a} \qquad (5.7)$$

where δ is the *CTOD*, V or *COD* is the crack opening displacement, r is the rotational factor that is approximately 0.44 for plastic components, a is the crack length and W is the specimen width.

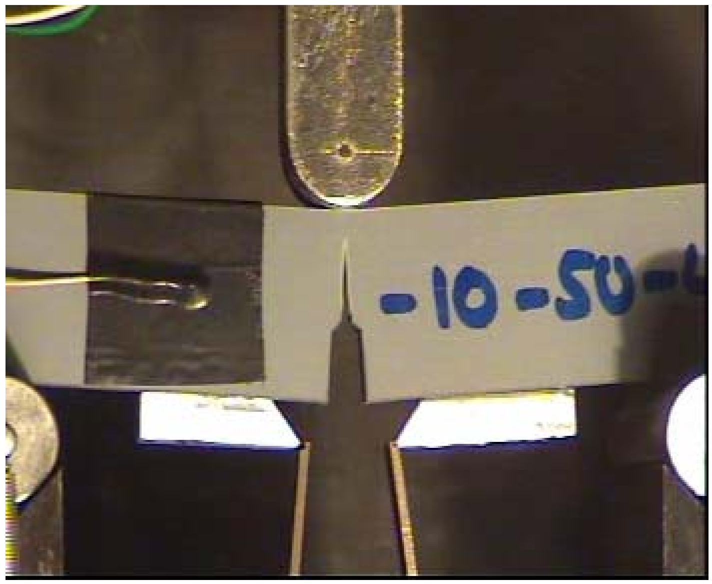


Figure 5.14 SENB specimen at 50 mm/min at -10^{o}C (Magnification =1.4x).

Figure 5.15 shows a photograph of SENB specimen with crack tip blunting at crosshead speed of 5 mm/min at the temperature of 23^oC. For this case, the measured value of crack length was 16.7 mm, the crack opening displacement, *COD*, was 8.6 mm and the calculated value of *CTOD* was 0.49 mm. Figure 5.16 illustrates the situation at 50 mm/min with a crack length of 17.3 mm, *COD* of 12.7 mm and *CTOD* of 0.53 mm. For a similar case at 500 mm/min, a = 17.3 mm, *COD* = 12.5 mm, *CTOD* was 0.52 mm, as shown in figure 5.17. At 23^oC, *CTOD* seems to remain constant as the crosshead speed increases from 5 to 500 mm/min. This is an indication that at 23^oC blunting may not be the cause for change in K_Q. It should be mentioned that the effect of crosshead speed on K_Q at this temperature is minimal as illustrated in figure 5.11. At high temperatures (50 and 70^oC), the crack tip blunting is found to increase monotonically with crosshead speed and fracture toughness also increases with crosshead speed. Crack tip blunting results didn't provide enough evidence for explaining the rate of loading effect on fracture toughness.

The photograph of SENB specimen with crack tip blunting at crosshead rate of 5 mm/min at 70^oC is shown in figure 5.18 for a crack length of 12.4 mm. The measured *COD* for this crack length is 7.6 mm and *CTOD* was 1.44 mm. Compared to figures 5.11 and 5.15, the crack tip at 70^oC is less sharp and the *CTOD* is larger. Therefore, the amount of crack tip blunting increases as the yield stress is decreased when the temperature increases.

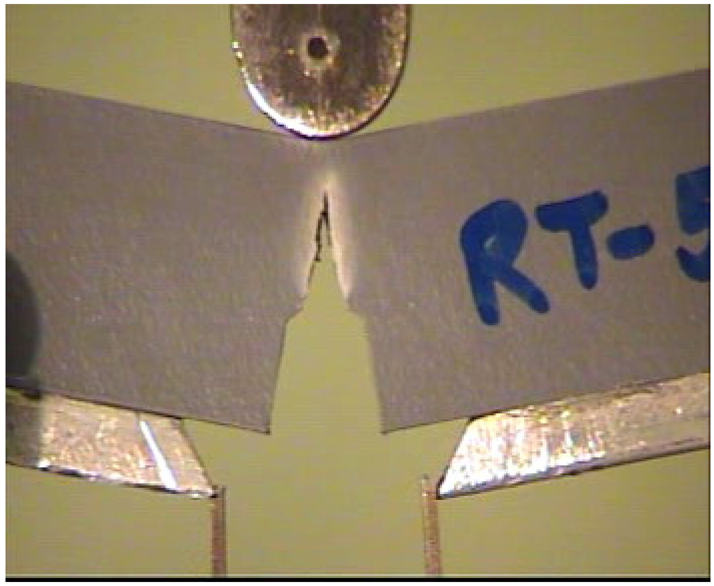


Figure 5.15 Crack tip blunting of SENB specimen at 5 mm/min at 23^{o}C (Magnification =2.4x).

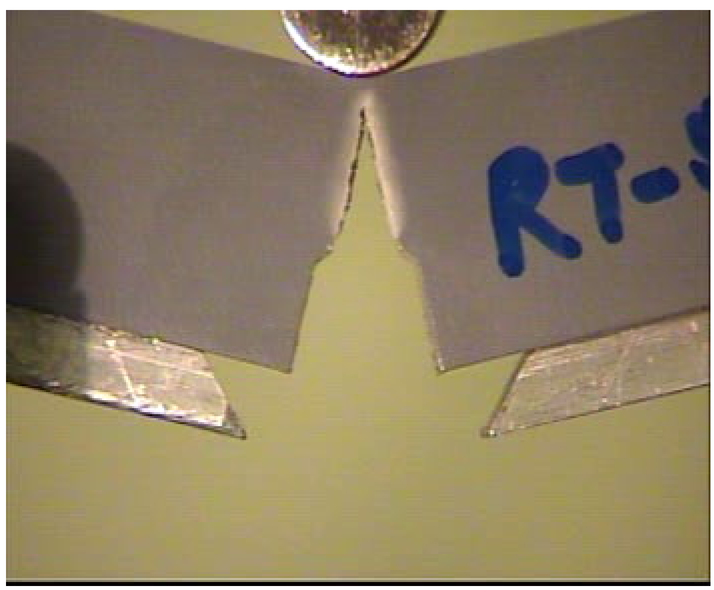


Figure 5.16 Crack tip blunting of SENB specimen at 50 mm/min at 23^{o}C (Magnification =2.4x).

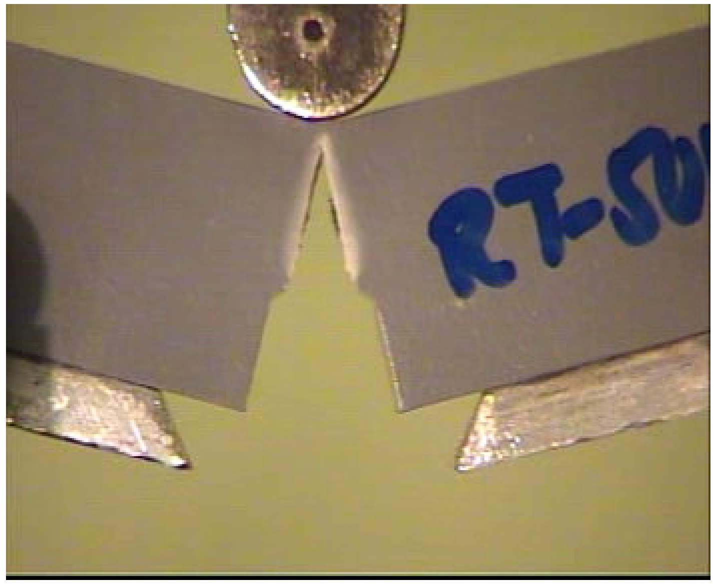


Figure 5.17 Crack tip blunting of SENB specimen at 500 mm/min at 23^{o}C (Magnification =2.4x).

Figure 5.18 Crack tip blunting of SENB specimen at 5 mm/min at 70^{o}C (Magnification =1.7x).

5.6 Crack Tip Plastic Zone

As with metals, a yield zone typically forms at the tip of a crack in polymers. In the case of shear yielding, the damage zone resembles the plastic zone in metals, because slip in metals and shear in polymers are governed by similar yield criteria. However, craze yielding produces a Dugdale-type strip yield zone ahead of the crack tip. Of the two yielding mechanisms in polymers, crazing is somewhat more likely ahead of a crack tip, because of the triaxial tensile stress state. Crazing is a highly localized deformation that leads to cavitations (voids formation) and strains on the order of 100% [53]. Usually, the width of a craze is of the order of 1 to 2 μm and it may grow to several millimeters in length depending on its interaction with other heterogeneities [54]. Shear yielding; however, can occur at crack tips in some polymers depending on the temperature and specimen geometry [49].

It should be noted that the crack propagation in polymers at high temperature is characterized by the formation of plastic zone or crazing. Crazing appears as a stress-whitened region, due to low refractive index. In the present investigation, stress-whitening was observed in most of the cases and more clearly at 70^{o}C as shown by the sequence of photographs in figure 5.19. The craze zone is formed perpendicular to the maximum principal stress.

In this study, the crack length and craze length were measured using video recording equipment during fracture toughness testing at crosshead rates of 5 and 50 mm/min. However, the crack length and craze length at 500 mm/min can not be measured because of very short period. The craze length was observed to increase with

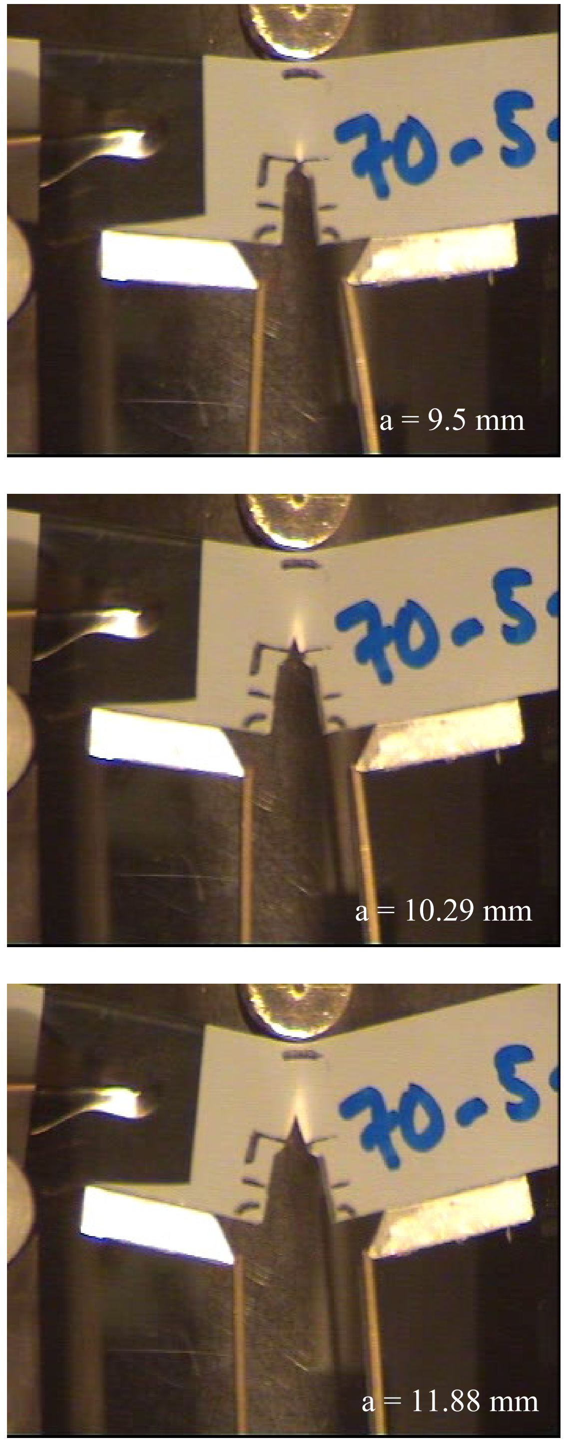


Figure 5.19 Photographs taken during crack growth showing the stress-whitened damage zone at 70°C and 5 mm/min for different crack lengths (Magnification =1.8x).

increasing crack length at all test temperatures at 5 mm/min. Figure 5.20 shows that craze length increases linearly with crack length and that the slope of the line increases with increasing temperature. More importantly, for a given crack length the craze length increases with temperature with a jump between 23 and 50^{o}C followed by a small increase between 50 and 70^{o}C. This may be the cause for the observed maxima in figure 5.12. At -10^{o}C, the craze zone length is small and the maximum value achieved is 1.9 mm for a crack length, a, of 15.5 mm, whereas for 70^{o}C crazes of up to 3.4 mm in length can be observed for 11.9 mm crack length. At 23^{o}C, the maximum measured craze length was 2.6 mm occurring at crack length of 14.5 mm. Similar behavior was found by Irfan-ul-Haq and Merah [44] for CPVC in fatigue crack growth tests.

Figure 5.21 illustrates the variation of craze zone length as a function of crack length at all test temperatures for a crosshead rate of 50 mm/min. It can be seen that the craze length increases linearly with crack length. Similar to earlier results the slope of the line increases with test temperature. For this crosshead speed, the value of craze length is lower than that for 5 mm/min at -10, 0 and 23^{o}C. For -10^{o}C and 50 mm/min, the increase in craze length is almost negligible due to the accumulated brittleness induced by low temperature and high crosshead speed. Similar to what was observed for 5 mm/min, a jump in the value of craze length is evident at 50^{o}C.

It is known that the craze length (ρ) is related to the yield stress of the material and the stress intensity factor at the crack tip [55]. Dugdale proposed a model that has been extensively applied to analyze polymer crack tip behavior:

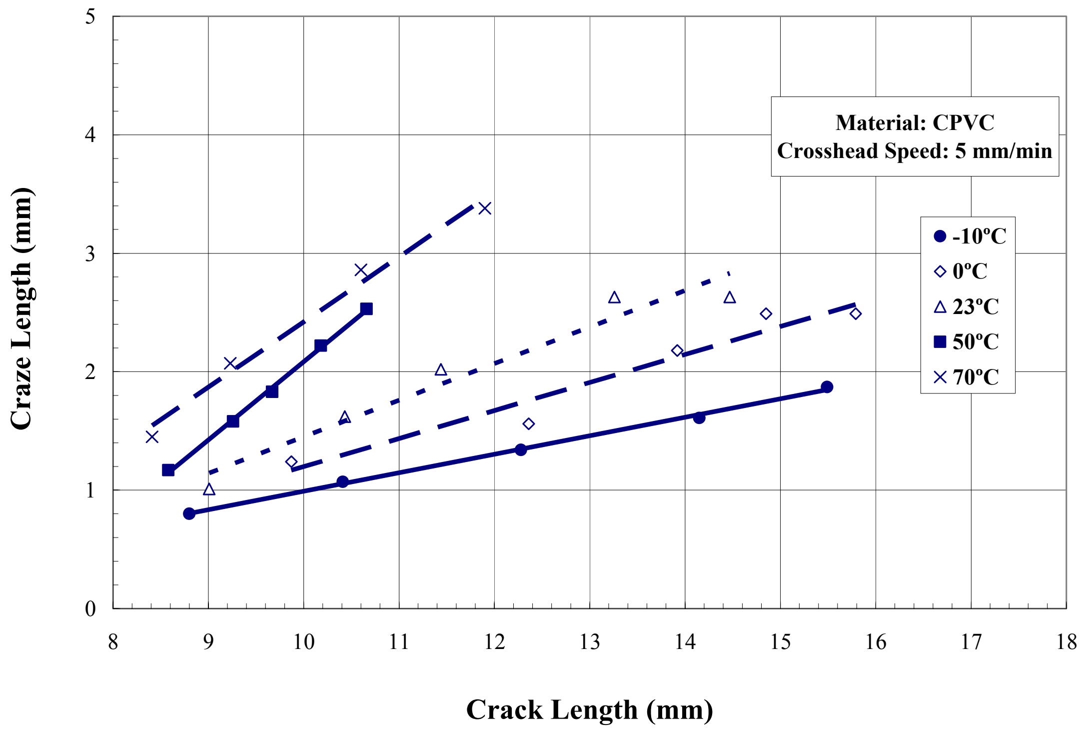


Figure 5.20 Craze length as a function crack length at different temperature at 5 mm/min.

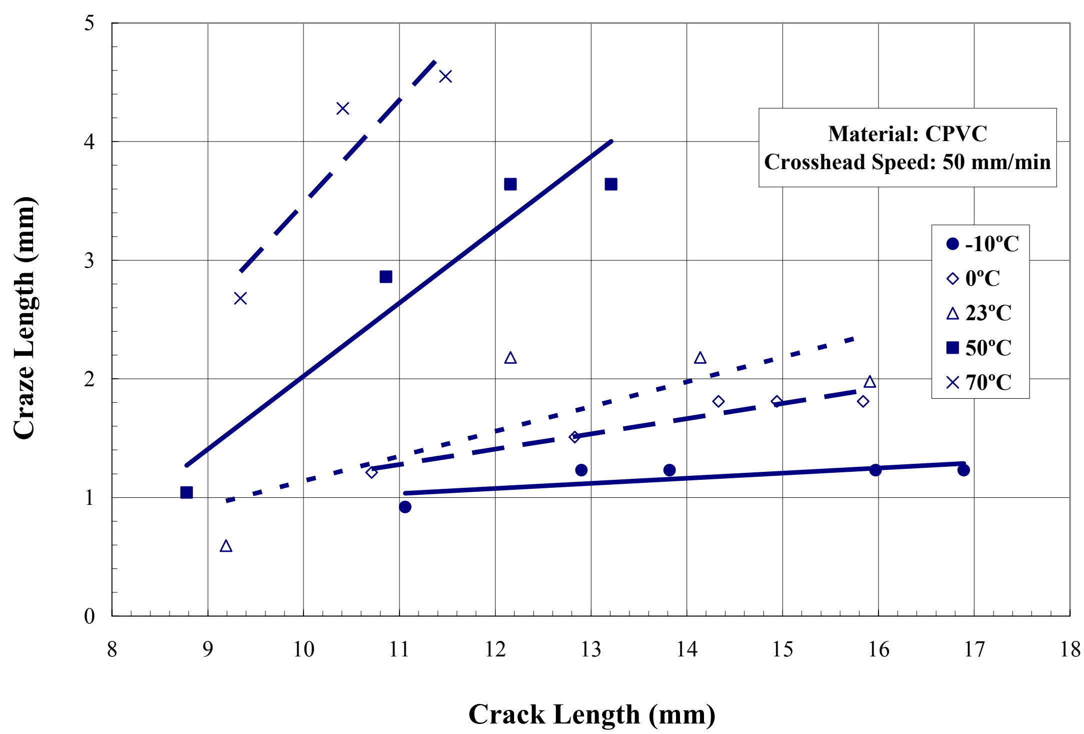


Figure 5.21 Craze length as a function crack length at different temperature at 50 mm/min.

$$\rho = \frac{\pi}{8}\left(\frac{K_I}{\sigma_y}\right)^2 \tag{5.8}$$

where σ_y is the yield strength and K_I is the stress intensity, given by:

$$K_I = \sigma\sqrt{\pi a} \tag{5.9}$$

using these two relations, one can relate craze length, ρ, to crack length, a, as follows:

$$\rho = \frac{\pi^2}{8}\left(\frac{\sigma}{\sigma_y}\right)^2 a \tag{5.10}$$

Equation 5.10 proves that similar to what was found from the present results craze length is linearly related to crack length. The constant C $= \frac{\pi^2}{8}\left(\frac{\sigma}{\sigma_y}\right)^2$ can be denoted as slope of the line representing the variation of craze length with crack length at a given temperature as shown in figure 5.20. Therefore, the craze length at the crosshead speed of 5 mm/min can be described as:

$$\rho = (0.0058T - 1.37)a \quad , T \text{ in Kelvin} \tag{5.11}$$

Similarly, the craze length at the crosshead speed of 50 mm/min can be expressed as:

$$\rho = (0.0104T - 2.73)a \quad , T \text{ in Kelvin} \tag{5.12}$$

It is known that the critical plastic deformation occurs at the crack tip during crack growth in polymers. The length of the critical plastic deformation zone at the crack tip, ρ_c, can be related to the fracture toughness, K_{IC}, by:

$$\rho_c = \frac{\pi}{8}\left(\frac{K_{IC}}{\sigma_y}\right)^2 \tag{5.13}$$

The development of plastic zone around the crack tip plays a very important role in controlling the measured fracture toughness and in determining the fracture mode whether brittle or ductile. The critical plastic zone and ductility factor calculated from equation 5.13 for PEI by Kim and Ye [23] showed increasing plastic zone size value with increasing temperatures.

It is useful to plot the plastic zone size as a function of crosshead speed at different temperatures as shown in figure 5.22. It is observed that the plastic zone size decreases with increasing crosshead speed at low temperatures (-10, 0 and 23^{o}C) and the fracture toughness also decreases between 50 and 500 mm/min. Above room temperature, the plastic zone size enhances with crosshead speed between 5 and 50 mm/min then decreases with further increasing crosshead speed. The plastic zone size increases with temperature for all crosshead speeds in the temperature range of -10 to 50^{o}C, as shown in figure 5.23. The maximum value of plastic zone size is reached around 50^{o}C (transition temperature from brittle to ductile fracture). Above 50^{o}C, the plastic zone size decreases with further increasing temperature. The fracture toughness was found (figure 5.12) to follow a behavior similar to that of plastic zone size with temperature. Therefore the dependence of plastic zone size on temperature and crosshead

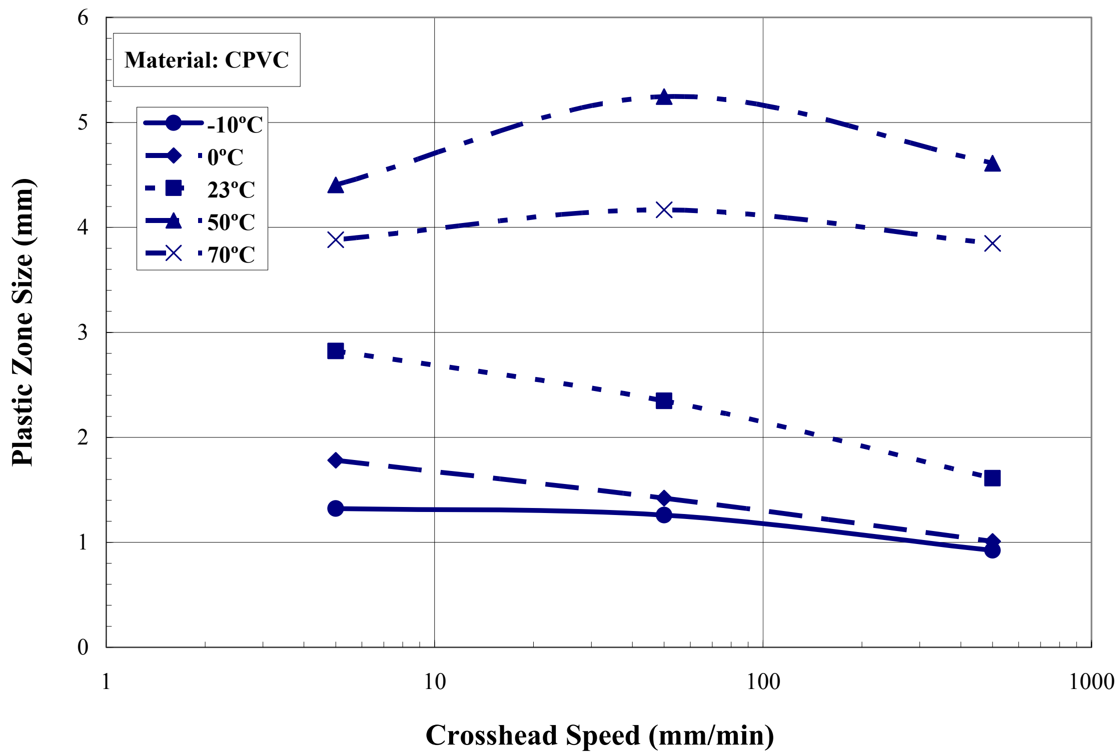


Figure 5.22 Plastic zone size as a function of crosshead speed at different temperatures.

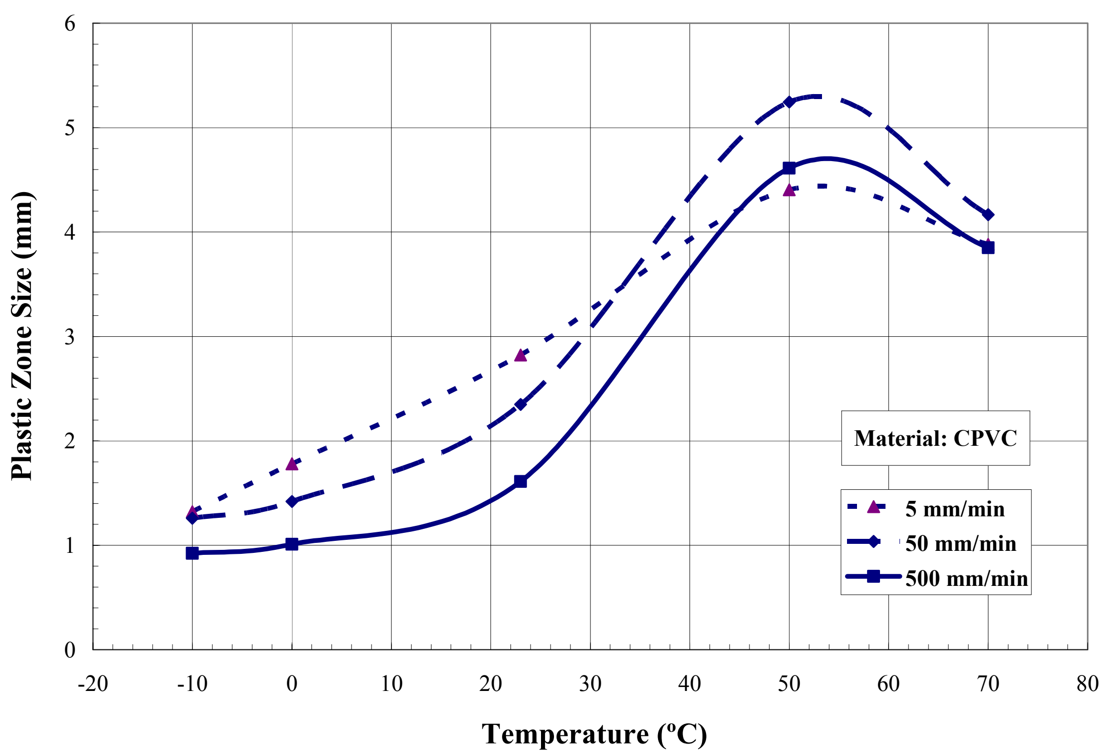


Figure 5.23 Plastic zone size as a function of temperature at different crosshead speeds.

speed can also be responsible for the dependence of fracture toughness on temperature and crosshead speed. This is in agreement with the plastic zone size behavior reported by Han et al. [14] for PEK-C.

5.7 Relationships between Fracture Toughness and Toughness Modulus

Toughness is a mechanical term which is a measure of the ability of a material to absorb energy up to fracture. The brittle material absorbs little energy, while a ductile material would require a large expenditure of energy in the fracture process. Maximum toughness, therefore, is achieved with an optimum combination of strength and ductility; neither high strength nor exceptional ductility alone provides for large fracture energy absorption [15].

The toughness modulus is calculated from the stress-strain curves. It is the area under stress-strain curve up to the point of fracture and can be expressed as:

$$Toughness = \int_0^{\varepsilon_f} \sigma d\varepsilon \tag{5.14}$$

where ε_f is the strain at fracture.

The average values of toughness modulus in terms of energy per unit volume (MJ/m^3) obtained from the measurement of the area under stress-strain curves at different crosshead speeds and temperatures (figures 4.1, 4.2 and 4.3) are presented in Table 5.8. At low temperatures (-10 and 0ºC), it can be seen that the toughness modulus decreases with increasing crosshead speed from 5 to 50 mm/min and then increases with further

Table 5.8 Average values of toughness modulus at different temperatures and crosshead speeds.

Temp. / v (mm/min)	-10ºC	0ºC	23ºC	50ºC	70ºC
5	3.08	3.15	3.29	3.27	3.72
50	2.29	2.92	6.88	6.55	10.76
500	3.63	4.59	7.17	7.09	10.54

increasing crosshead speed. However, it increases with crosshead speed at room temperature and above. The toughness modulus increases slowly with increasing temperature in the temperature range of -10 to 50ºC for all crosshead speeds. However, it increases rapidly between 50 and 70ºC.

The quantitative relationships between the measured value of fracture toughness and toughness modulus are illustrated in figure 5.24. At low crosshead speed (v = 5 mm/min), it can be observed that the value of fracture toughness seems to increase with increasing toughness modulus up to 50ºC then it decreases. Similar general behavior is observed at moderate and high crosshead speeds (v = 50 and 500 mm/min).

5.8 Fractography Analysis

The examination of fracture surfaces (fractogrphy) has been used widely in the study of metals, glass and polymers. In metals, it is generally possible to tell from the appearance of the surfaces where fracture has originated, in which direction it has traveled and what has been its general nature (eg. ductile or brittle). The fracture surfaces of polymeric materials yield similar data and their study is of great practical importance as well as scientific interest [1]. On the basis of these enlightening information, material and design engineers are better able to improve overall component design through changes in composition and internal microstructure of materials [6].

Most polymers like metals yield at sufficiently high stresses. While metals yield by dislocation motion along slip planes, polymers can exhibit either crazing or shear yielding as they do not contain crystallographic planes, dislocations and grain boundaries.

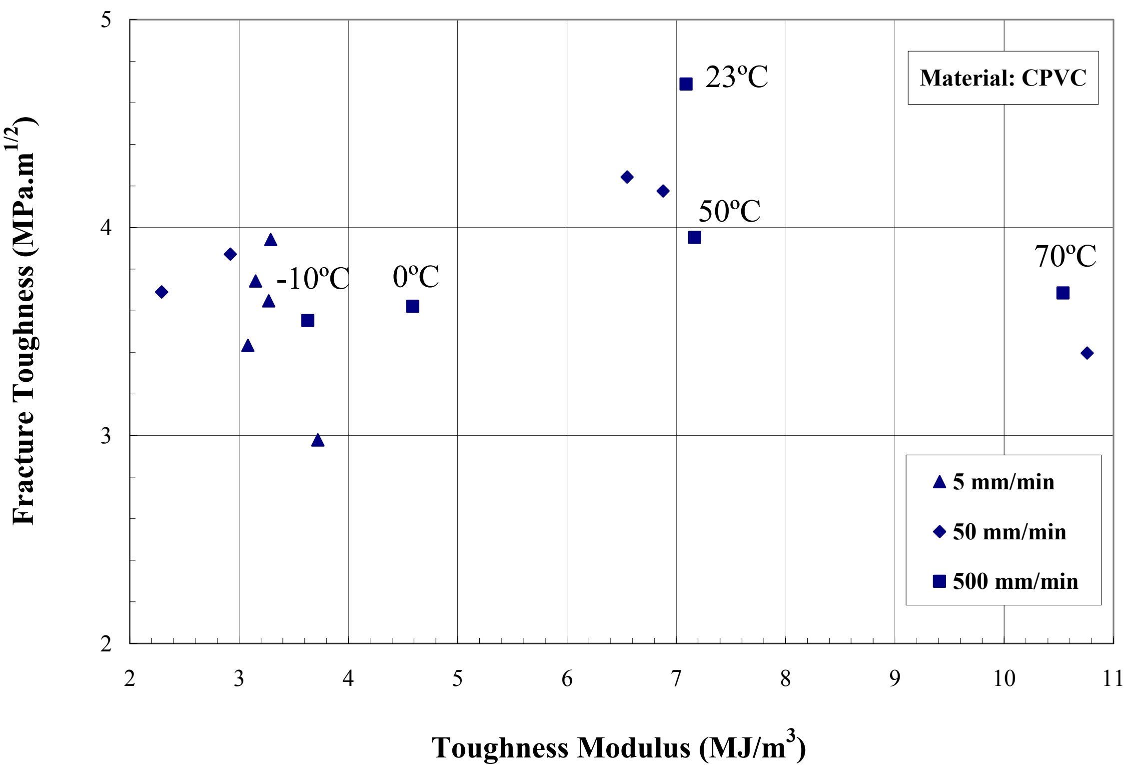
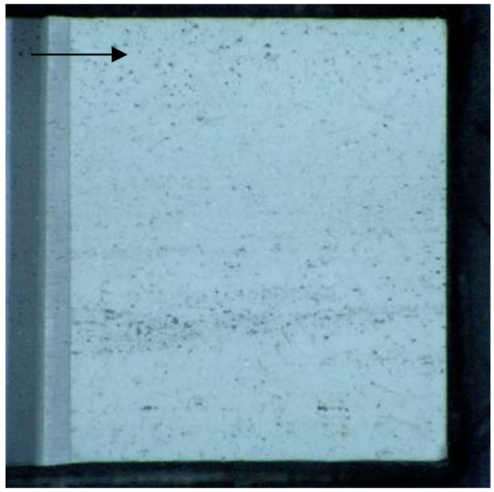


Figure 5.24 Fracture toughness versus toughness modulus at different temperatures and crosshead speeds.

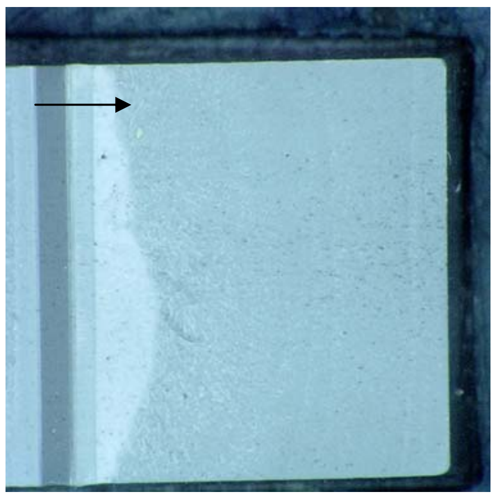
Shear yielding in polymers resembles plastic flow in metals, at least from a continuum mechanics viewpoint. Molecules slide with respect to one another when subjected to a critical shear stress. Crazing and shear yielding are competing mechanisms; the dominant yielding behavior depends on molecular structure, stress state and temperature [56].

In order to investigate the mechanisms of fracture in CPVC pipe material, fracture surfaces of SENB specimens tested at different crosshead speeds and temperatures were prepared and analyzed using digital camera system (Pixera) and scanning electron microscope (SEM). Figures 5.25-5.29 show the macrofractographs of fracture surfaces at different crosshead speeds and temperatures with the arrows showing the direction of crack propagation. It is evident from these fractoghraphs that the fracture surface roughness decreases with increasing crosshead speed at all temperatures. This is an indication of increasing material brittleness with increase in crosshead speed. The reduction of fracture surface area increases with increasing crosshead speeds at high temperatures (50 and 70^{o}C). This necking-like behavior is produced by increase in ductility and presence of 3-D state of stress.

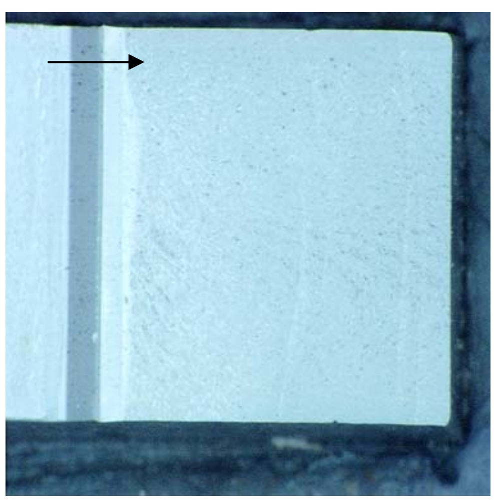
In addition, it is noticed that the fracture surface roughness increases with temperature up to 50^{o}C because of increasing plastic zones or crazing then decreases between 50 and 70^{o}C and so does the fracture toughness. The dominant fracture mechanism at high temperature is found to be crazing characterized by rough fracture surfaces.



(a) 5 mm/min (Mag. =18.7x)

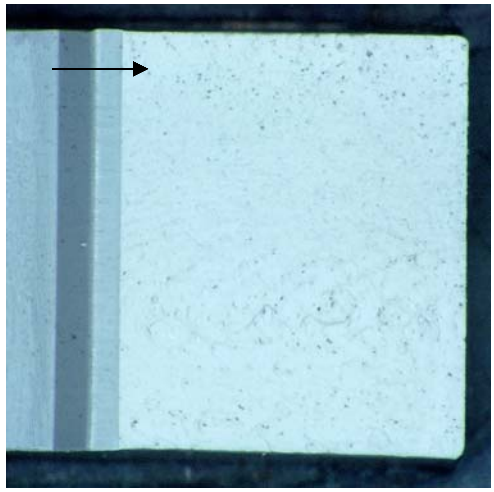


(b) 50 mm/min (Mag. =18.8x)

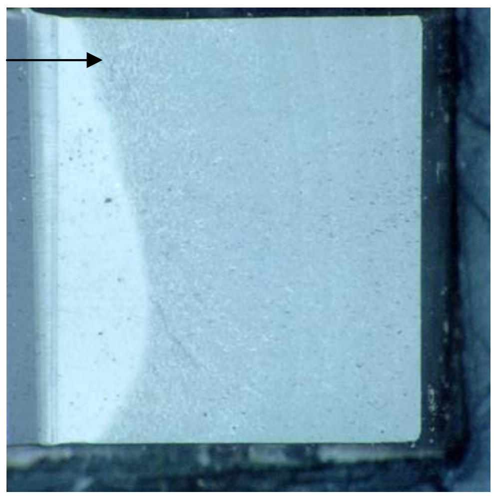


(c) 500 mm/min (Mag. =17.5x)

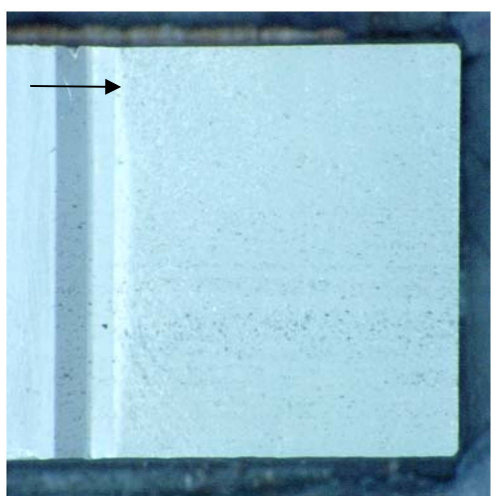
Figure 5.25 Macrofractographs at -10^{o}C for different crosshead speeds.



(a) 5 mm/min (Mag. =18.5x)

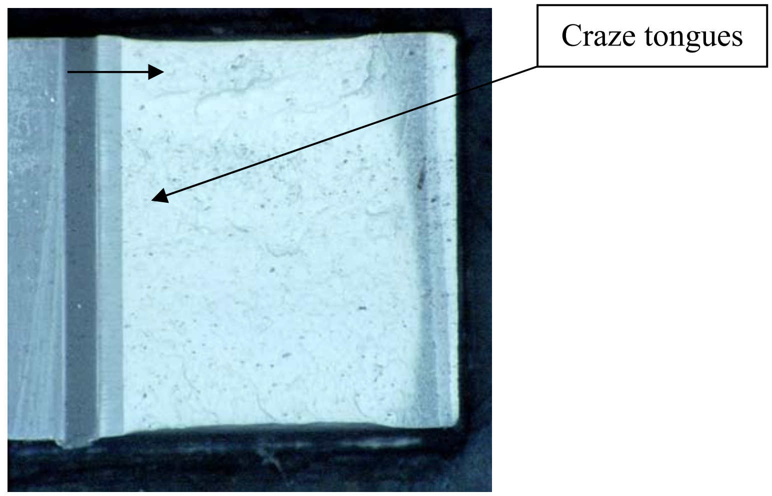


(b) 50 mm/min (Mag. =18.9x)

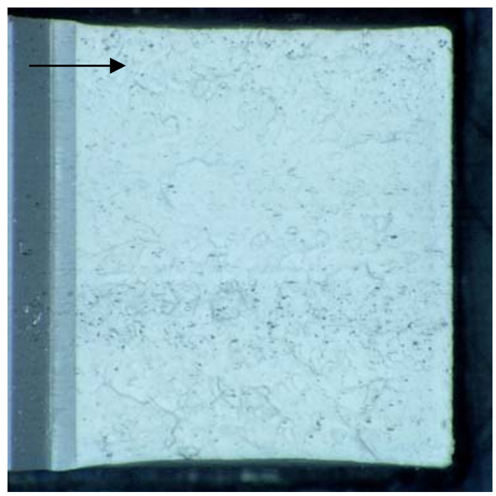


(c) 500 mm/min (Mag. =18.6x)

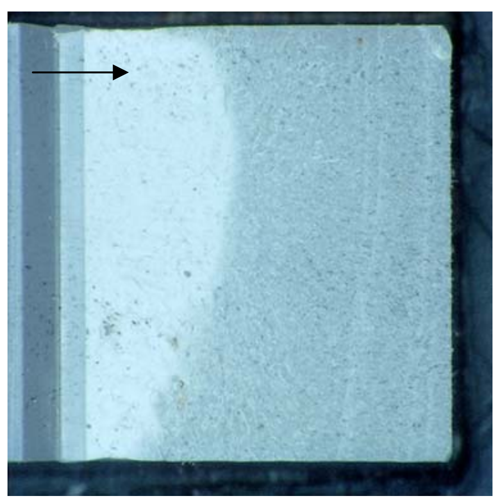
Figure 5.26 Macrofractographs at $0^{o}C$ for different crosshead speeds.



(a) 5 mm/min (Mag. =16.9x)

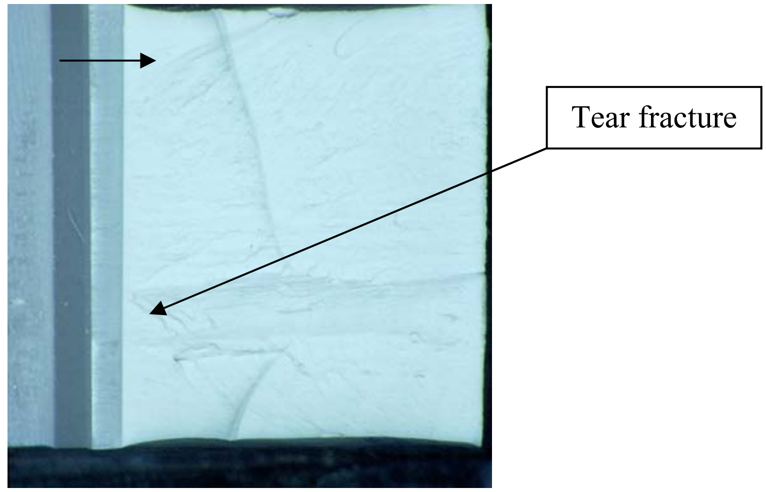


(b) 50 mm/min (Mag. =19.1x)

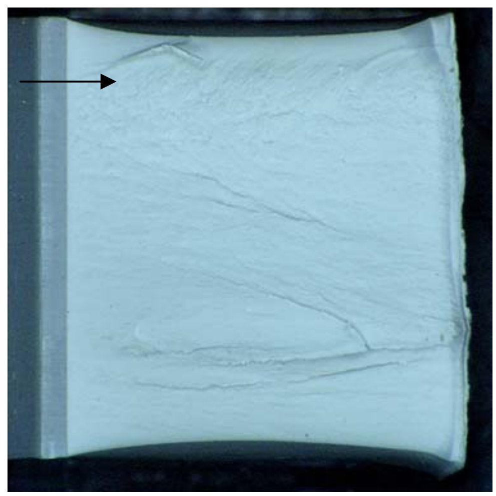


(c) 500 mm/min (Mag. =19.3x)

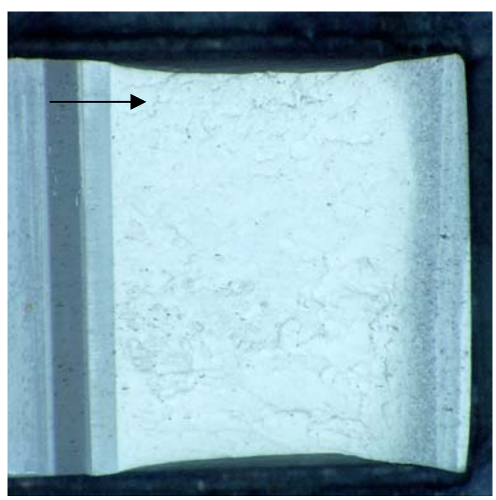
Figure 5.27 Macrofractographs at 23^{o}C for different crosshead speeds.



(a) 5 mm/min (Mag. =18.5x)

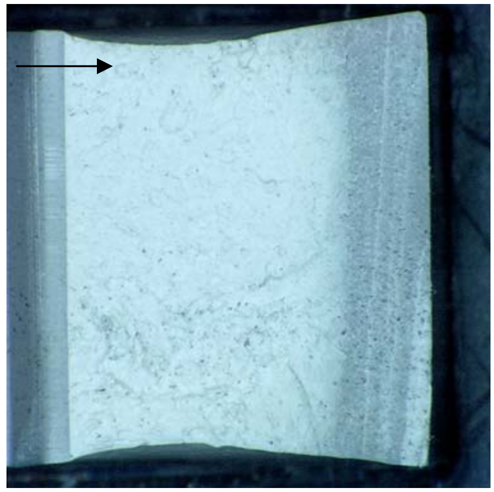


(b) 50 mm/min (Mag. =20x)

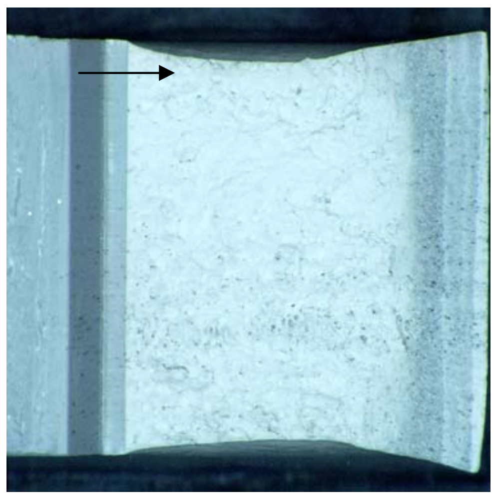


(c) 500 mm/min (Mag. =18.4x)

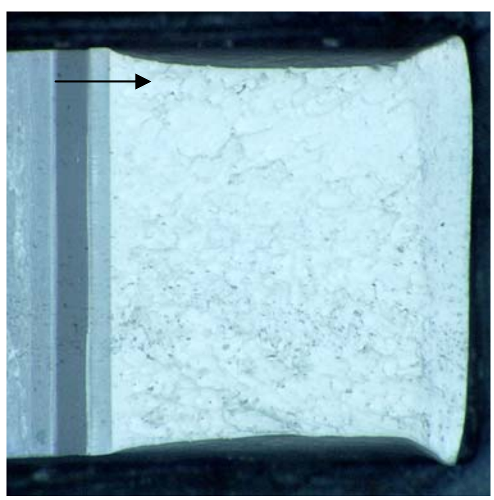
Figure 5.28 Macrofractographs at 50^{o}C for different crosshead speeds.



(a) 5 mm/min (Mag. =19x)



(b) 50 mm/min (Mag. =18.3x)



(c) 500 mm/min (Mag. =18.5x)

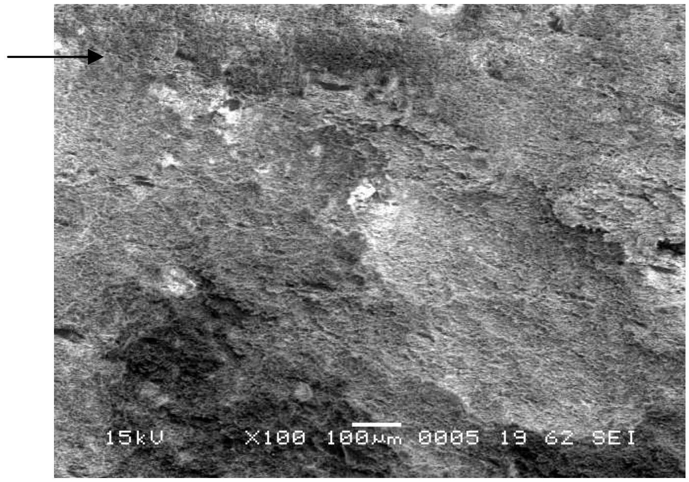
Figure 5.29 Macrofractographs at 70^{o}C for different crosshead speeds.

To further investigate the fracture mechanisms, higher magnification fractographs are taken. Figure 5.30 represents SEM fractographs of fracture surfaces of specimens tested at 5 mm/min at 0 and 70^{o}C. It is evident that the fracture surface at 70^{o}C is rougher than that at 0^{o}C. At room temperature and above, it is observed that the reduction of fracture surface area increases with increasing temperature because of increasing ductility.

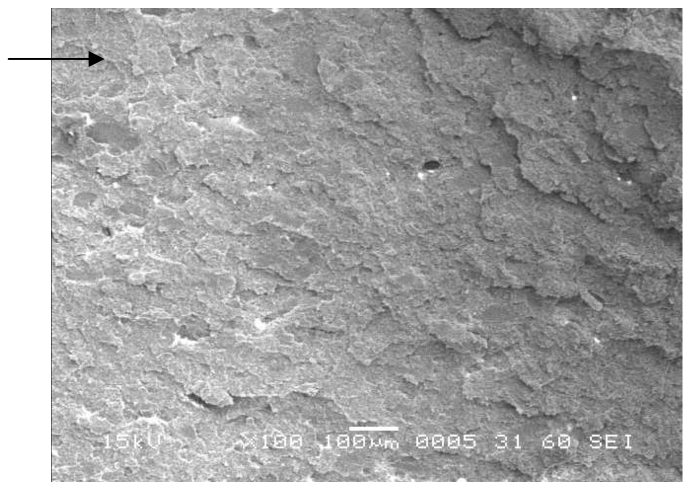
The fracture surface of specimens tested at low temperatures (-10 and 0^{o}C) fractured in a brittle unstable manner showing flat fracture surface at low crosshead speed (5 mm/min). At moderate and high crosshead speeds (50 and 500 mm/min), it is observed that the fracture surface is rough at the onset of the crack and decreases with increasing crosshead speed. At these temperatures (-10 and 0^{o}C), the operative dominant fracture mechanism was found to be shear yielding by Saghir [34] and Irfan-ul-Haq and Merah [44] on CPVC pipe fitting material.

Significant craze tongues are observed on fracture surface of specimens tested at room temperature at 5 mm/min, as shown in figure 5.27. These tongues and roughness decreased with increasing crosshead speed. Also, the reduction of fracture surface area decreased with increasing crosshead speed, decreasing from 2.1% of area reduction at 5 mm/min to 0.14% at 500 mm/min.

Figure 5.28 shows that at 50^{o}C (brittle-to-ductile temperature), the crack propagation displays tear fracture behavior at low and moderate crosshead speed. However at 500 mm/min, craze tongues are observed without any tear fracture behavior. The reduction of fracture surface area increases with increasing crosshead speeds. The percentage of area reduction increases from 2.52% at 5 mm/min to 5.5% at 500 mm/min.



(a) 0^{o}C (Center of fracture surface)

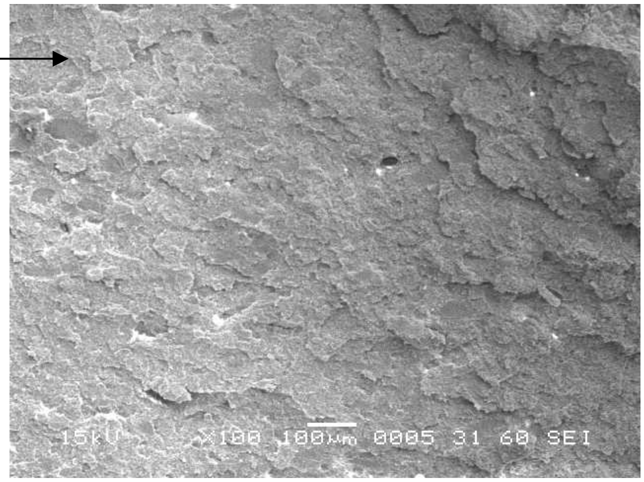


(b) 70^{o}C (Top of fracture surface near crack initiation)

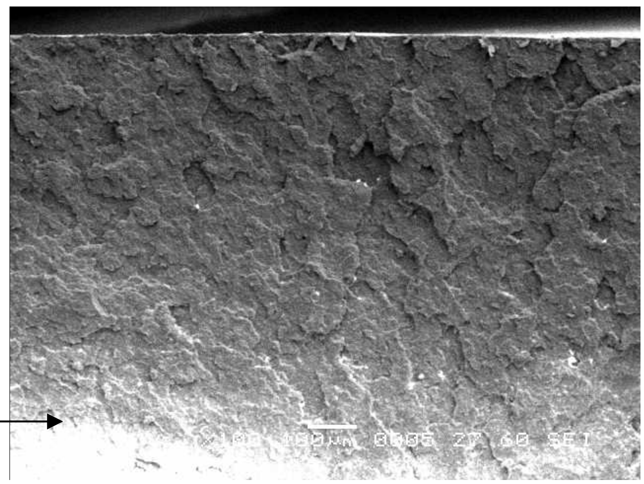
Figure 5.30 SEM fractographs at 5 mm/min at 0 and 70^{o}C (Mag. =100x).

Mizutani [17] observed change in fracture surface mechanism which was unstable crack propagations (stick-slip behavior) of PMMA in the temperature range of 70 to 90^{o}C below the glass transition temperature (T_g = 107^{o}C) at a crosshead rate of 0.5 mm/min. Similar crack growth behavior has also been observed Ye et al. [33] for TPX at T = 60 ^{o}C, below T_g (81^{o}C), at 0.5 mm/min.

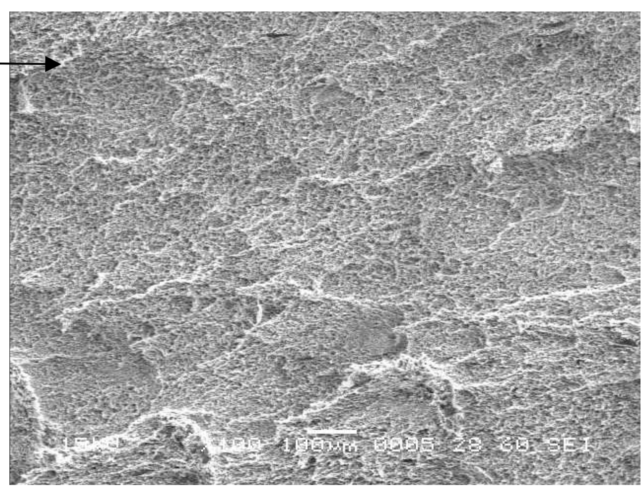
When the temperature is further increased to 70^{o}C, the crack growth becomes stable and the tear fracture is not observed, as shown in figure 5.29. It is noticed that craze tongues and roughness decrease with increasing crosshead speed. The fracture toughness also decreases at this temperature. The reduction of fracture surface area increases with crosshead speed, increasing from 5.12% of area reduction at 5 mm/min to 9.14% at 500 mm/min. SEM fractographs of figure 5.31 show fracture surfaces of SENB specimens tested at 70^{o}C for different crosshead speeds. At low crosshead speed, plastic deformation with stress whitening on the fracture surface is visible (figure 5.31a). This is typical evidence of ductile fracture. However, the fracture surface at high crosshead speed is much more brittle without any ductile fracture mechanism (figure 5.31c). At high temperatures crazing is the dominant fracture mechanism.



(a) 5 mm/min (Top of fracture surface near crack initiation)



(b) 50 mm/min (Top of fracture surface near crack initiation)



(c) 500 mm/min (Center of fracture surface)

Figure 5.31 SEM fractographs at 70^{o}C for different crosshead speeds (Mag.=100x).

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

The effects of strain rate and temperature on fracture toughness and tensile properties of CPVC pipe material were studied. In this chapter, the important conclusions of this thesis work are summarized. Recommendations for future research work in this field are also included.

6.1 Conclusions

In this study, the effects of strain rate and temperature on tensile properties and fracture toughness of commercial CPVC pipe material have been studied. The crosshead speeds and temperatures considered in this study are 5, 50 and 500 mm/min and -10, 0, 23, 50 and 70^{o}C, respectively. Tensile tests were performed on dog bone specimens while the fracture toughness tests were performed using single-edge-notch bending specimens. Fractography and LEFM concepts were used in analyzing fracture toughness results.

6.1.1 Tensile Properties

The following conclusions are obtained from the investigation of the effects of strain rate and temperature on tensile properties:

- The yield stress and elastic modulus increased as the crosshead speed increased at all test temperatures; higher rigidity and material strength are promoted by higher strain rates.
- The yield stress and elastic modulus decreased with increasing temperature at all crosshead speeds. This CPVC material is stiffer at low temperature because of reduced molecular mobility. At high crosshead speed, there is insufficient time for the material to respond to stress with large-scale viscoelastic deformation or yielding.
- The fracture strain remained almost constant with crosshead speed at room temperature and below. At 50 and 70^{o}C, an enhancement of fracture strain occurred with increasing crosshead speed between 5 and 50 mm/min then stabilized with further increasing crosshead speed.
- The fracture strain increased slowly in the -10 to 23^{o}C range and rapidly between 50 and 70^{o}C. Ductile fracture is the governing mechanism in the 50 to 70^{o}C region. At 70^{o}C, considerable necking is observed to occur at 50 and 500 mm/min.

6.1.2 Fracture Toughness

The value of fracture toughness at different crosshead speeds and temperatures was determined. The following conclusions can be drawn:

- The plane strain fracture toughness condition was satisfied at -10^{o}C for all crosshead speeds whereas the criterion was met for some dimensions in most cases at 0^{o}C. The size criterion was not met for all dimensions in all cases for the temperatures of 23, 50 and 70^{o}C because of large plastic zone. It should be noted that this study was limited by the thickness of pipe.
- The value of fracture toughness increased slightly with crosshead speed between 5 and 50 mm/min at low temperatures (-10, 0 and 23^{o}C). However for the range of 50 to 500 mm/min, fracture toughness reduced slightly. The fracture toughness enhanced with crosshead speed at high temperatures (50 and 70^{o}C). The dependence of fracture toughness on crosshead speed was mainly the result of plastic zone size which was found to control this effect. Crack tip blunting results didn't provide enough evidence for explaining the rate of loading effect on fracture toughness.
- At low crosshead speed (v = 5 mm/min), the value of fracture toughness increased with temperatures below ambient temperature. It decreased slightly with increasing temperature between 23 and 70^{o}C. It increases continuously with temperature at moderate and high crosshead rates (v = 50 and 500 mm/min) up to the brittle to ductile transition temperature (50^{o}C). Then, it dropped with further increasing temperature. Similar general behaviors were observed for the crack tip blunting and craze length with temperature. The temperature dependence of plastic

zone size may also be responsible for the temperature dependence of fracture toughness.

- The relationships between the measured value of fracture toughness and toughness modulus were explained. It was observed that the value of fracture toughness seemed to increase with increasing toughness modulus up to 50ºC then it dropped for all crosshead speed.
- The fracture surface roughness reduced with increasing crosshead speed at all temperatures because of increasing material brittleness. The reduction of fracture surface area increased with crosshead speed at high temperatures (50 to 70^{o}C). In addition, it was noticed that the fracture surface roughness increased with temperature up to 50^{o}C because of increasing plastic zone or crazing then decreased between 50 and 70^{o}C and so did the fracture toughness. At room temperature and above, it was observed that the reduction of fracture surface area increased with temperature because of enhanced ductility.

6.2 Recommendations

Following are some of the recommendations for any future research that can be carried out on CPVC and similar polymers:

- To have a better representation of the effect of strain rate and temperature on fracture toughness and tensile properties of CPVC, lower crosshead speeds and higher temperatures can be considered. More tests in the studied range at temperatures in the transition zone (23 to 50^{o}C) will help identifying the brittle to ductile transition temperature.
- The effect of outdoor exposure on fracture toughness and tensile properties of CPVC may yield useful data for construction companies.
- The effect of specimen size on fracture toughness can be investigated to have valid plane-strain fracture toughness at high temperatures.
- When large plastic zones are formed the linear elastic fracture toughness K does not remain valid, so elastic plastic fracture mechanics concepts can be used to study fracture toughness. An important parameter is the J-integral. Critical J values for polymers exhibit less size dependence than K_Q values.
- Time dependent fracture properties such as creep and stress relaxation can be sought.

REFERENCES

1. Andrews E. H., “Fracture in Polymers”, American Elsevier Publishing Company, Inc., 1968.

2. Lampman S., “Characterization and Failure Analysis of Plastics”, ASM International, 2003.

3. “Boedeker Plastics: PVC & CPVC Datasheet”, www.boedeker.com.

4. “Modern Plastics Encyclopedia”, 1994, pp B-10-B-14.

5. Odian George, “Principles of Polymerization”, 2nd Edition, A Wiley-Interscience Publication, John Wiley & Sons, Inc., 1981.

6. Irfan-ul-Haq M., “Crack Propagation in CPVC at Different Temperatures”, M.S. Thesis, KFUPM, December 2000.

7. Scott Brann and Michelle Knight, “Consider CPVC for Process Applications”, Chemical Engineering Process, Dec 1994, pp 36-41.

8. Saudi Plastic Products Company Limited (SAPPCO)- Dammam, www.sappco-dammam.com.

9. Callister W. D., “Materials Science and Engineering: an Introduction”, John Wiley & Sons, Inc., 3rd Edition, 1994.

10. Ward I. M., “Mechanical Properties of Solid Polymers”, 2nd Edition, Wirly-Interscience Publication, 1983.

11. Anderson T. L., “Fracture Mechanics Fundamentals and Applications”, CRC Press LLC, 2nd Edition, 1995.

12. Swallowe G. M., “Mechanical Properties and Testing of Polymers an A-Z Reference”, Polymer Science and Technology, 1999.

13. Dowling N. E., “Mechanical Behavior of Materials”, Prentice Hall, 2nd Edition, 1993.

14. Yanchun Han, Yuming Yang, Binyao Li and Zhiliu Feng, “Temperature and strain-rate dependence of fracture toughness of phenolphthalein polyether ketone”, Journal of Materials Science, Volume 30, 1995, pp 3658-3661.
15. Hertzberg R. W., “Deformation and Fracture Mechanics of Engineering Materials”, 4th Edition, John Wiley & Sons Inc., 1996.

16. Langford G., Whitney W. and Andrews R. D., Material Research, Lab. Research Report No. R63-49, MIT School of Engineering, Cambridge, 1963.

17. Mizutani Kiyoshi, "Temperature dependence of fracture toughness of poly(methyl methacrylate)", Journal of Materials Science Letters, Volume 6, 1987, pp 915-916.

18. Tordjeman P., Teze L., Halary J. L. and Monnerie L., "On the Plastic and Viscoelastic Behavior of Methylmethacrylate-Based Random Copolymers", Polymer Engineering and Science, Volume 37, No. 10, October 1997, pp 1621-1632.

19. Parvin M. and Williams J. G., Journal of Materials Science, Vol. 10, 1975, pp 1883-1888.

20. Kitagawa M., "Power Law Relationship Between Yield Stress and Shear Modulus for Glassy Polymers", Journal of Polymer Science: Polymer Physics Edition", Vol 15, 1977, pp 1601-1611.

21. Bronnikov S. V., Vettegren V. I. and Frankel S. YA., "Description of Thermal and Mechanical Properties of Drawn Polymers Over a Wide Temperature Range", Polymer Engineering and Science, Volume 32, No. 17, Mid-September 1992, pp 1204-1208.

22. Stokes V. K. and Nied H. F., "Solid Phase Sheet Forming of Thermoplastics-Part I: Mechanical Behavior of Thermoplastics to Yield", Journal of Engineering Materials and Technology, Volume 108, April 1986, pp 107-112.

23. Ki-Young Kim and Lin Ye, "Effects of thickness and environmental temperature on fracture behavior of polyetherimide (PEI)", Journal of Materials Science, Volume 39, 2004, pp 1267-1276.

24. Fernando P. L. and Williams J. G., Polymer Engineering Science, Vol. 20, 1980, pp 215-220.

25. Chan M. K. V. and Williams J. G., Polymer Engineering Science, Vol. 21, 1981, pp 1019-1026.

26. Jia Nanying and Kagen Val A., "Mechanical Performance of Polymaides with Influence of Mositure and Temperature-Accurate Evaluation and Better Understanding", Plastic Failure Analysis and Prevention, USA, 2001.

27. Povolo F., Schwartz G. and Hermida E. B., "Stress Relaxation of PVC Below the Yield Point", Journal of Polymer Science, Volume 34, 1996, pp 1257-1267.

28. Bauwens-Crowet C., Bauwens J. A. and Homes G., Journal of Polymer Science, A2, pp 735, 1969.

29. Che M., Grellmann W. and Seidler Sabine, "Crack Resistance Behavior of Polyvinylchloride", Journal of Polymer Science, Volume 64, No. 6, May 1997, pp 1079-1090.

30. Hitt D. J. and Gilbert M., "Tensile properties of PVC at elevated temperatures", Materials Science and Technology, Volume 8, August 1992, pp 739-745.

31. Green J. L. and Petty C. A., "Relationship Between Strain Rate, Temperature, and Impact Failure Mechanism for Poly(Vinyl Chloride) Poly(Ethylene Terephthalate)", Polymer Engineering and Science, Volume 38, No. 1, January 1998, pp 194-203.

32. Vicent P. I., Plastics, London, Vol. 27, January 1962.

33. Lin Ye, Chao-Ting Yuan and Yiu-Wing Mai, "Effect of Rate and Temperature on Fracture Behavior of TPX (Poly-4-Methyl-1-Pentene) Polymer", Polymer Composites, Volume 19, No. 6, December 1998, pp 830-836.

34. Saghir S. F., "Effect of Frequency on Crack Propagation in CPVC and HDPE at Different Temperatures", M.S. Thesis, KFUPM, January 2004.

35. Merah N., Irfan-ul-Haq M. and Khan Z., "Temperature and weld-line effects on mechanical properties of CPVC", Journal of Materials Processing Technology, Volume 142, 2003, pp 247-255.

36. Williams J. G., "Fracture Mechanics of Polymers", Ellies Harwood Limited, John Wiley & Sons, USA, 1984.

37. Mandell J. F., Darwish A. Y. and McGarry F. J., "Time and Temperature Effects on the Fracture Toughness of Rigid Poly(vinylchloride) Pipe Materials", Polymer Engineering and Science, Volume 22, No. 13, September 1982, pp 826-831.

38. Marshall G. P., Coutts L. H. and Williams J. G., Journal of Materials Science, Vol. 9, 1974, pp 1409-1419.

39. Karger-Kocsis J. and Friedrich K., "Temperature and strain-rate effects on the fracture toughness of poly(ether ether ketone) and its short glass-fiber reinforced composite", Polymer, Volume 27, November 1986, pp 1753-1760.

40. Pecorini T. J. and Hertberg R. W., "The fracture toughness and fatigue crack propagation behavior of annealed PET", Polymer, Volume 34, No. 24, 1993, pp 5053-5062.

41. Marshall G. P., Culver L. E. and Williams J. G., International Journal of Fracture, Vol. 9, 1973, pp 295-309.

42. Parvin M. and Williams J. G., Journal of Materials Science, Vol. 11, 1976, pp 2045-2050.

43. Nimmer R. P., Weiss K., McGuire J., Takemori M. and Morielli T., “Engineering Application of Linear Elastic Fracture Mechanics for a Ductile Polymer Part”, Proceedings of the Society of Plastics Engineers Annual Technical Meeting (Atlanta, GA), Society of Plastics Engineers, 1988.

44. Irfan-ul-Haq M. and Merah N., “Effect of Temperature on Fatigue Crack Growth in CPVC”, Journal of Pressure Vessel Technology, Volume 125, No. 1, February 2003, pp 71-77.

45. Merah N., Al-Qahtani T., Khan Z. and Zainalabidine, “Effect of strain rate and temperature on fracture toughness of CPVC”, Proceeding of 1st IMS International Conference, UAE, January 2004.

46. “Standard Test Method for Plane-Strain Fracture Toughness and Strain Energy Release Rate of Plastic Materials”, American Society for Testing and Materials, Designation: D5045-93.

47. “Standard Test Method for Tensile Properties of Plastics”, American Society for Testing and Materials, Designation: D638-01.

48. Berry B. W., “Fracture Toughness”, ISI Publication 121,The Iron and Steel Institute, 1968.

49. Cayard M., “Fracture Toughness Testing of Polymeric Materials”, Ph.D. Dissertation, Texas A&M University, College Station, TX, September 1990.

50. “Standard Test Method for Fracture Toughness of Metallic Materials”, American Society for Testing and Materials, Designation: E399-90.

51. Atkin A. G. and Mai Y. W., Elastic and Plastic Fracture, Eills Horwood/Wiley, Chichester, United Kingdom, 1985.

52. Kinloch A. J. and Williams J. G., Journal of Materials Science, Vol. 15, 1980, pp 987.

53. Bucknall, C.B., “Toughened Plastics”, Applied Science Publishers, London, 1977.

54. Donald A. M. and Kramer E. J., "Effect of Molecular Entanglements on Craze Microstructure in Glassy Polymers", Journal of Polymer Science: Polymer Physics Edition, Vol. 27, 1982, pp 889-909.

55. Bernal-Lara T. E., Hu Y., Hiltner A. and Baer E., "Stepwise Fatigue Crack Propagation in Poly(Vinyl Chloride)", ANTEC 2003.

56. Hertzberg R.W. and Manson J.A., "Fatigue of Engineering Plastics", Academic Press, USA, 1980.

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